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SUPPLEMENT 8

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A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

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Statement A
Approved for Public Release

DIRECTORATE OF MATERIALS AND PROCESSES
AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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FOREWORD

This report was prepared by Miss Junie J. Banks and Mrs. Donna J. Tate, Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Technical reports published by the Directorate of Materials and Processes during the period 1 July 1960 - 30 June 1961 are abstracted herein. Reports on research conducted by Directorate of Materials and Processes personnel as well as that conducted on contract are included.

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ABSTRACT

These reports cover basic and applied research in the materials area being conducted by the Metals and Ceramics, Nonmetallic Materials, Physics, Manufacturing Technology and Applications Laboratories of the Directorate of Materials and Processes.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER



D. A. SHINN, Chief
Materials Information Branch
Applications Laboratory
Directorate of Materials & Processes

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I. Technical Reports and Technical Notes 1 July 1960 - 30 June 1961

CERAMICS, CERMETS AND GRAPHITE

CERAMICS AND CERMETS

WADC TR 58-12, Part III, OTS Release

May 1960

SUBJECT: HIGH-TEMPERATURE ELECTRICAL INSULATING INORGANIC COATINGS
ON WIRE

INVESTIGATOR: David L. Wilcox, Clifton G. Bergeron, Paul F. Schwarzlose,
Arthur L. Friedberg

CONTRACT: AF 33(616)-3943, University of Illinois

ABSTRACT: The program on high-temperature electrical insulation on
wire, in its third and final year, covered the work on flexibility of coated
wire, coating trials on clad copper wires, firing procedure for two coats of
58C on copper wire, vitreous silica and boron nitride mill additions to glass
coating 58C, and vitreous coatings sheathed with organics. Also, as a part
of the last years effort, test data were obtained on high-temperature insulated
wire supplied by various manufacturers.

Flexibility studies indicated the ratio of coating thickness to
wire diameter and the cooling cycle were important factors affecting flexi-
bility of the coating. It was demonstrated that a heat treatment of the coating
may increase coating flexibility. Contrary to what was expected, coatings on
inconel-clad copper wire did not show an increase in flexibility over coatings
on plain copper wire.

The results of coating trials on inconel-clad, stainless
steel-clad (oxalloy), nickel-clad (Kulgrid), silver-clad, and aluminum-clad
copper wires are given. A firing study of coating 58C resulted in the optimum
firing procedure for two coats of 58C on copper wire.

It was demonstrated that additions of vitreous silica and
boron nitride to the mill formula of coatings 58 and 58C enhance the electrical
properties of the resulting coating.

Sheathing of vitreous coatings with organics showed promise
as a method of retaining the flexed vitreous coating on wire. It was thought
that in service the organic coating may burn off at a temperature where the
vitreous coating could be capable of sealing itself.

Electrical tests of flexed coated specimens, indicated that
room temperature dielectric constant and dissipation factor were not sensitive
to the first fine cracks which appeared.

Manuscript released November 1961 as an ASD Technical Report.

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WADC TR 58-452, Part III, OTS Release

November 1960

SUBJECT: METAL FIBER REINFORCED CERAMICS
INVESTIGATOR: J. R. Tinklepaugh, B. R. Goss, W. R. Hoskyns, J. H. Connor,
D. D. Button
CONTRACT: AF 33(616)-5898, College of Ceramics, Alfred University
ABSTRACT: The flexural properties of a ceramic-metal fiber system were studied and it was found that the metal fiber does assume a part of the load which is to some degree in proportion to the relative elasticity moduli of the ceramic and metal. The ceramic fails when its strength is exceeded but the composite does not fail until the metal fibers are broken or pulled out of the ceramic. The test data for the alumina-molybdenum and alumina-mullite-molybdenum systems were extended to 3000°F. Hafnium oxide was found to have desirable characteristics for use in a composite system.

WADC TR 59-102, Part II

October 1960

SUBJECT: DEVELOPMENT AND EVALUATION OF INSULATING TYPE CERAMIC COATINGS
INVESTIGATOR: H. Leggett, R. L. Johnson, E. W. Blocker, E. D. Weisert
CONTRACT: AF 33(616)-5441, The Marquardt Corporation
ABSTRACT: A metal reinforced, insulating ceramic coating system has been developed which will successfully withstand temperatures greater than 4000°F. The composite is of the gross type and it consists of a chemically bonded zirconia or zirconia chromia phase bonded at low temperatures, reinforced with and anchored to a coated refractory metal. The composite system exhibits low thermal conductivity and excellent thermal shock resistance.

The developed macro-composite utilizes the desirable properties of refractory metallic structures by providing both insulation and oxidation protection. The metallic reinforcement, protected in the same manner, strengthens the ceramic layer and serves to anchor it to the basic metal structure.

WADC TR 59-300, Part II

October 1960

SUBJECT: RESEARCH AND DEVELOPMENT SERVICES LEADING TO THE CONTROL OF ELECTRICAL PROPERTIES OF MATERIALS FOR HIGH TEMPERATURE RADOMES
INVESTIGATOR: Leon M. Atlas, Hikaru Nagao
CONTRACT: AF 33(616)-5929, Armour Research Foundation
ABSTRACT: Alumina ceramics having less than 100 ppm total impurities and densities up to about 3.67 g/cm³ were fabricated by firing cold pressed calcined alumina hydrate in air at temperatures up to about 1960°C. These high purity specimens were supplemented by a series of deliberately contaminated ceramics prepared by introducing known concentrations of Si, Ti, Mg, Ca, Fe, and Cr into the batch. The loss tangents and dielectric constants (k') of these

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discs were measured at MIT in the region 10^2 to 10^6 c/s and 25 to 500°C. Statistical analysis of the resulting data was then carried out at ARF by multiple regression methods. At 10^6 c/s and 500°C $\tan \delta$ was linearly related to impurity concentration with a multiple correlation coefficient of 0.93. Si ions caused the greatest rise of $\tan \delta$, and Mg, Ti, Ca, Cr, and Fe had progressively decreasing effects. In contrast, the linear correlation coefficient at 10^2 c/s was lower than that obtained from a semi-logarithmic function, which gave a value of 0.80.

Loss tangent and dielectric constant data were also used to calculate the conductivities of the specimens at 10^5 c/s, and the activation energies of conduction at 500°C were then deduced from plots of $\log \sigma$ vs $1/T$. In almost every case a value in the range 1.2 to 1.6 ev was obtained. Comparison with trapping energies calculated from glow curves suggests that electrons associated either with Si or Ti impurity sites were the predominant charge carriers. However, in discs doped with Mg^{2+} , the activation energy rose to 2 ev. Glow curve calculations indicate that Mg^{2+} creates traps with a depth of about 2.9 ev; therefore, Mg ions contributed significantly to the reservoir of charge carriers.

Replotting the $\tan \delta$ and k' data into curves of k'' vs. frequency reveals small peaks which have been attributed to interfacial polarization controlled by grain boundaries. Using the known electrical measuring potential and the microscopically determined grain diameters, an estimated carrier mobility of $11-15 \text{ cm}^2/\text{volt sec}$ at 25°C was calculated.

With the objective of further reducing the small positive temperature coefficient of k' of alumina, ceramics were prepared with a compensating titanate phase. Mixtures containing 90% Al_2O_3 and 10% $CaTiO_3$ were found to have an invariant dielectric constant of 11.4 to 11.7 up to 250°C at 10^7 c/s. At this frequency, the loss tangent of the two phase ceramic at 500°C is lower than that of Pyroceram 9606.

WADC TR 59-316, Part II, OTS Release

May 1960

SUBJECT: MECHANISM OF WEAR OF NONMETALLIC MATERIALS
INVESTIGATOR: C. H. Riesz
CONTRACT: AF 33(616)-5962, Armour Research Foundation
ABSTRACT: A fundamental study was made of the wear mechanism of nonmetallic materials at high temperatures. Single-crystal sapphire was examined in a friction apparatus made for studies up to 2200°C. A sapphire ball loaded at 22, 50, and 127 g was passed over a sapphire plate at 0.0053 cm/sec at 29-921°C. Both slider and plate were oriented such that the C₀-axis was normal to the direction of traverse.

WADC TR 59-316, Part II (Continued)

With cleaned, as-received materials coefficient of friction values were scattered. Prior heat treatment at 900-925°C reduced scatter and gave peak values of 0.25-0.30 near 600°C. The results suggest that the surfaces of the sapphire were imperfect. Observations by other investigators on variation of modulus of rupture appear to have a common basis.

The wear areas on both ball and plate were 6 microns wide, and subsurface changes extended to 30 microns. Electron microscopy inspection suggests that extremely high surface temperatures occurred coincidently with the friction process, but direct evidence of an adhesion mechanism is inconclusive.

WADC TR 59-432, Part II, OTS Release

July 1960

SUBJECT: REFRactory INORGANIC MATERIALS FOR STRUCTURAL APPLICATIONS
INVESTIGATOR: Harry A. Pearl, John M. Nowak, Joseph C. Conti
CONTRACT: AF 33(616)-5930, Bell Aircraft Corporation
ABSTRACT: A process was developed for making both small and large, simple and complex curvature geometric shapes from honeycomb ceramic sandwich constructions. Final fired sections had apparent densities as low as 26 lb/ft³.

A simple mechanical method was developed for continuous fabrication of ceramic corrugated ribbon or sheet for making honeycomb cores. The core is flexible and easily machinable in its prefired condition. The skins are as easy to handle as paper.

Alumina-silica paper impregnated with an alumina-silica slurry was used as a refractory model system for developing the fabrication techniques. The paper originally acts as a carrier but finally becomes an integral part of the fired refractory. The fabrication and processing methods are adaptable to other refractory materials possessing higher strength and temperature stability.

Other types of ceramic and metal-ceramic composites were investigated and evaluated.

WADC TR 59-526, Part II, OTS Release

August 1960

SUBJECT: PROTECTIVE COATINGS FOR REFRACTORY METALS
INVESTIGATOR: Clifton G. Bergeron, Victor J. Tenney, Arthur L. Friedberg, Doris M. Maroney, Robert D. Shannon
CONTRACT: AF 33(616)-5734, University of Illinois
ABSTRACT: Various ceramic coating systems for tungsten metal were developed and evaluated. These included silicide coatings, ceramic crystals in a glass matrix, zirconia-clad coatings, vapor-deposited SiO, and recrystallizing glasses. The system considered protective for tungsten at temperatures

WADC TR 59-526, Part II (Continued)

of 3000°F or higher, from a practical viewpoint, was a coating consisting of 35% glass and 65% zircon.

Tungsten wire specimens coated with this glass-zircon coating were protected for ten hours at 3000°F. A study of the nature of the degradation of this coating tested at 3000°F as a function of time was carried out based on microstructural observations and X-ray diffraction analysis of the reaction products. The protective lifetime of the coating was related to the degree of attack of the coated tungsten metal as indicated by interfacial roughness and tungsten oxide formation, and to the progressive decrease in the thickness of the coating under test.

WADC TR 59-744, Volume III

July 1960

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME III. Screening Test Results and Selection of Materials

INVESTIGATOR: Frank M. Anthony, Harry A. Pearl

CONTRACT: AF 33(616)-6034, Bell Aircraft Corporation

ABSTRACT: The purpose of this contract was to investigate the feasibility of utilizing available heat resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This particular volume presents the results of the preliminary portion of the material evaluation portion of the program. Material requirements were established for this specific application. In order to assess the suitability of a given material, knowledge of many material characteristics is required. Of primary importance are oxidation resistance, high thermal emissivity, high thermal conductivity, low thermal expansion, reproducible strength characteristics, and fabricability. Other characteristics which must be considered are specific heat, modulus of elasticity, erosion characteristics and density. Over 100 technical reports were reviewed, and approximately 50 suppliers and fabricators were contacted to obtain data on materials which might be suitable for leading edge applications at 2500°F to 3000°F. A total of 106 materials were considered including 22 refractory metals, 30 protective coatings and 54 refractory non-metals.

From the review of available materials it was apparent that much of the information required for assessing suitability was lacking. A screening test program was initiated, therefore, to fill the gaps in available data and to provide consistent sets of data upon which to base intelligent selections. The methods used in choosing the materials to be screened considered availability and the complex interrelation among design and material parameters. From the 106 candidate materials three metals, five coating systems and seven non-metals were subjected to screening tests. Determination of oxidation resistance, thermal emissivity, thermal conductivity, thermal expansion, mechanical strength and modulus of elasticity, were made from room temperature to 2700°F.

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Using the data obtained experimentally and the methods developed for assessing suitability, the .5% titanium alloy of molybdenum, Chromalloy W-2 coating, Durak MG coating and siliconized ATJ graphite were found to be the most promising materials for the specific application. Consequently these materials were selected for further evaluation.

WADC TR 59-744, Volume IV, OTS Release

October 1960

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME IV. Thermal Properties of Molybdenum Alloy and Graphite

INVESTIGATOR: I. B. Fieldhouse, J. I. Lang, H. H. Blau, Jr.

CONTRACT: AF 33(616)-6034, Armour Research Foundation, Arthur D. Little, Inc.

ABSTRACT: The purpose of this contract was to investigate the feasibility of utilizing available heat resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This particular volume presents the results of measurements of the thermal conductivity, specific heat, linear thermal expansion, and emittance of a 0.5% titanium alloy of molybdenum, and of siliconized ATJ graphite as a function of temperature. Emittance measurements were made on coated and uncoated materials.

WADD TR 60-54, OTS Release

May 1960

SUBJECT: CHARACTERISTICS GOVERNING THE FRICTION AND WEAR BEHAVIOR OF REFRACTORY MATERIALS FOR HIGH TEMPERATURE SEALS AND BEARINGS

INVESTIGATOR: Lewis B. Sibley, Arthur E. Mace, Daniel R. Grieser, C. Malcolm Allen

CONTRACT: AF 33(616)-3995, Battelle Memorial Institute

ABSTRACT: An investigation of the basic factors involved in the wear and friction of ceramics, cermets, and high-temperature alloys sliding at speeds of 100 to 200 fps and temperatures from 500 to 1000°C (1000°F to 1800°F) has been made. Bearing pressures covered a range of 5 to 50 psi. Statistical correlation of measured wear rates with friction and material properties indicates that, at these temperatures, the wear rate of ceramics and cermets may be approximately described by the following relationship:

$$\text{Wear rate} \propto \frac{\mu}{R^{1.25} D^{0.75}},$$

where μ is the coefficient of friction, R is the thermal-stress-resistance factor, and D is the thermal diffusivity of the mated material on which wear

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predominates, which is usually the material with the lowest thermal-stress resistance.

A mechanism of wear has been evolved based on the above correlation and on the experimental study of friction and wear surface-temperature fluctuations using special transducers and color motion-picture photography. The predominant wear mechanism in the high-speed sliding of ceramic and cermet materials appears to involve formation of hot spots at asperity contacts and subsequent fracture of the material near these hot spots as a result of thermal stresses. In this situation the wear rate is influenced both by the configuration of the rubbing parts and by the thermal-stress-resistance properties of the materials. When one of the mating surfaces is interrupted, the total wear is greater. Wear tends to predominate on parts with interrupted wear surfaces, such as slider bearings, in comparison with their continuous mating surfaces or ring surfaces, such as in face seals, and on ceramic and cermet materials with low thermal-stress-resistance factor and low thermal diffusivity.

Promising commercial materials for high-temperature dry sliding bearings, gas bearings, and seals include Al_2O_3 -Cr-Mo cermets, SiC ceramics, and TiC-Ni-Mo cermets. One experiment with a $\text{SiC}-\text{Si}_3\text{N}_4$ ring stator mated against a TiC-Ni-Mo-NbC rotor, as in a face seal, was operated for a total of 20 hours up to 1000°C (about 1800°F), 200-fps sliding speed, and 20-psi unit load with only slight surface damage and a total wear of 6.7 mils, most of which occurred on the rotor.

WADC TR 60-108, OTS Release

April 1960

SUBJECT: INVESTIGATION OF SINTERABLE POWDERS AND PROPERTIES OF BERYLLIA CERAMICS
INVESTIGATOR: James E. Johnson, A. K. Smalley, Winston H. Duckworth
CONTRACT: AF 33(616)-6238, Battelle Memorial Institute
ABSTRACT: Information was developed on the effects of processing variables and microstructure on the fracture strength of ceramics made from sinterable oxide powders. The characteristics of high strength being associated with low porosities and small average crystal sizes was observed in ceramics of both MgO and BeO , but no quantitative correlation was found. Highest strengths were obtained when the time and temperature of sintering were sufficient for densification above about 96 per cent of theoretical, provided that the sintering time and temperature was insufficient to give crystal sizes larger than the smallest observed, 5-6 microns for BeO ceramics and about 10 microns for MgO ceramics.

In the case of BeO powder prepared by pyrolysis of high-purity sulfate, calcining to an intermediate temperature (1700 - 1800°F) gave the powder that densified most readily when compacted and sintered, but one that

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did not have the greatest tendency toward crystal growth. Thus, this most sinterable powder produced the strongest BeO ceramic.

The lack of quantitative correlations between bulk density, average crystal size, and strength, together with appreciable scatter in strength values, indicated that strength was affected by some factor or factors other than these two. Localized areas of crystal growth found in BeO specimens may have had an influence.

WADD TR 60-109, Part I

June 1960

SUBJECT: DEVELOPMENT AND EVALUATION OF SOLID PROPELLANT ROCKET MOTOR CASE INSULATING MATERIALS SYSTEMS
INVESTIGATOR: James D. Batchelor, N. Vasileff, Stewart W. McCormick, Eugene L. Olcott
CONTRACT: AF 33(616)-6316, Atlantic Research Corporation
ABSTRACT: This report summarizes the results of the first year of a program designed to improve the capabilities of rocket motors which use end-burning grains through: (1) development of laboratory test procedures to be used as criteria for the evaluation of insulating materials, and (2) development of materials for application as motor case insulation. A literature survey and stress analysis of the motor insulation system were conducted. Procedures have been established to measure the elastic properties of tube specimens and the thermal conductivity of composite materials. The pyrolysis of resins and resin-bonded ceramic materials has been studied using a laboratory image furnace, and the gaseous and liquid decomposition products have been collected and analyzed.

The materials development work has included the comparison of insulating behaviors, erosion resistance, and charring rates of several resins in a torch test. It also includes a study of the carbon yield from resins and composite materials. With respect to insulation ability, resins which yield a high percentage of volatile products usually appear best in the torch test, but no simple correlation is evident. The yield of carbon necessary to produce coherent char structure can be investigated by measuring the physical and thermal properties of typical charred residues.

An extension of this program during the coming year, under Contract AF 33(616)-6831, will include specialized motor firings to test materials under service conditions; the laboratory work will be continued to correlate laboratory and motor data.

WADD TR 60-124, OTS Release

April 1960

SUBJECT: DEVELOPMENT OF NON-OXIDIC REFRACtORY FOAMS
INVESTIGATOR: Ian M. Logan, Douglas C. Wise, John J. McGahan,
Carl vonDoenhoft
CONTRACT: AF 33(616)-6294, Carborundum Company
ABSTRACT: Fundamental technology has been developed for the preparation of non-oxidic refractory foams of controlled pore diameter, spacing, and continuity.

Foaming procedures used in making commercially available foam plastics were employed to produce the resin-type foams which formed the basis for the final refractory foams.

It was shown that foam structures can be varied as to strength, density and pore size by varying the proportions of resins and solvents used to generate the foams. Lesser controls were also possible by varying the temperature and pressure during the foaming process. Good control and predictable results were obtained.

Foams were prepared of the following materials: carbides of titanium, tungsten, molybdenum, tantalum, and columbium; borides of titanium and of zirconium; and silicides of tungsten and of molybdenum.

WADD TR 60-143, OTS Release

July 1960

SUBJECT: RESEARCH STUDY TO DETERMINE THE PHASE EQUILIBRIUM RELATIONS OF SELECTED METAL CARBIDES AT HIGH TEMPERATURES
INVESTIGATOR: R. T. Dolloff
ABSTRACT: The work here reported is the result of an investigation of phase equilibria in the ternary system, silicon-boron-carbon. Techniques for extending the high temperature range of measurements have been developed. Accurate phase diagrams for the silicon-carbon and the boron-carbon binaries have been obtained and details of the ternary system have been determined in the high carbon content region. The data were obtained by high temperature differential thermal analysis and by photoelectric thermal analysis, both supplemented by metallographic, X-ray and chemical techniques.

Results for the boron-carbon binary system indicate that boron melts at $2130 \pm 10^{\circ}\text{C}$ and that B_4C has a congruent melting point of $2470 \pm 20^{\circ}\text{C}$. Boron and B_4C form a eutectic at $2080 \pm 20^{\circ}\text{C}$, whereas carbon and B_4C form a eutectic at $2390 \pm 20^{\circ}\text{C}$.

Results for the silicon-carbon binary indicate a eutectic between silicon and SiC at $1402 \pm 5^{\circ}\text{C}$. SiC melts incongruently at $2540 \pm 40^{\circ}\text{C}$. The peritectic point is located at 27 per cent carbon.

Results for the ternary system indicate a eutectic between B_4C , SiC and carbon at $2250 \pm 20^{\circ}\text{C}$. No compound formation has been observed in the high carbon region of the ternary system.

WADD TR 60-184, OTS Release

August 1960

SUBJECT: EFFECT OF BASIC PHYSICAL PARAMETERS ON ENGINEERING PROPERTIES OF INTERMETALLIC COMPOUNDS
INVESTIGATOR: D. L. Wood, J. H. Westbrook
CONTRACT: AF 33(616)-6144, General Electric Research Laboratory
ABSTRACT: To facilitate subsequent studies of the nature of the brittleness in intermetallic compounds, a method for producing sound, uniform, and reproducible test specimens has been devised and an investigation made of the effects of basic physical parameters on the mechanical properties.

Tensile test specimens of Bi_2Tl and AgMg , produced directly by extrusion, have provided information applicable to future studies of the properties of NiAl , a material whose melting point and oxidation resistance render it not impractical as an alloy base should a solution to the ductility problem be found.

A pronounced yield point is found in AgMg ; high strain rate sensitivity of the yield stress has been observed and the effects of grain size, composition, and test temperature have been documented. Specimens will withstand loading only at very slow strain rates; after yielding occurs, however, rapid strain rates may be employed. With total elongations of more than 50 per cent, the material is ductile under previously brittle conditions. Measurement of the yield stress as a function of both strain rate and temperature enables a calculation of the activation energy for the yielding process.

WADD TR 60-244, Part II, OTS Release

February 1961

SUBJECT: CONTINUOUS FILAMENT CERAMIC FIBERS
INVESTIGATOR: Edward H. Girard
CONTRACT: AF 33(616)-6246, The Carborundum Company
ABSTRACT: A number of refractory materials were examined to determine their possible usefulness as materials for crucibles for melting, fining, and drawing high-silica glass compositions. Niobium di-boride gave the most promising results of the materials examined.

Time and temperature relationships as well as variations in batch composition were investigated in an effort to improve premelt characteristics. The addition of vanadium pentoxide was particularly effective. It had been conceived that a well-fined premelt should be a better starting material for fiber drawing than an unreacted batch composition. Fiber drawing studies, however, showed no improvement in either the drawing characteristics or in the physical characteristics of fiber when well-fined premelt was used.

November 1960

SUBJECT: THE INFLUENCE OF HIGH PRESSURES AND HIGH TEMPERATURES
ON TRANSFORMATIONS AND COORDINATION IN CRYSTALLINE AND
VITREOUS CERAMIC MATERIALS

INVESTIGATOR: W. F. Claussen, R. C. DeVries, J. D. MacKenzie

CONTRACT: AF 33(616)-6295, General Electric Research Laboratory

ABSTRACT: The effect of high pressures and temperatures on densification of vitreous silica and germania and on the polymorphism of crystalline Al_2SiO_5 was investigated by using the high-pressure techniques developed for diamond synthesis.

Germania glass of a density approximately equivalent to that of the quartz-form germania was made by this technique. The highest density achieved for vitreous silica was 2.559, as compared to 2.648 for crystalline quartz. A shift of IR reflection bands at 1110 cm^{-1} for SiO_2 and 895 cm^{-1} for GeO_2 suggested that the densification of the glasses is a result of the decrease of the M-O-M angle, but the over-all spectra for both compressed vitreous SiO_2 and GeO_2 are unchanged from the correspondingly less dense glasses.

As a result of the study of the system Al_2SiO_5 at high temperatures and pressures, it was established that the high-pressure form, kyanite, melts incongruently above $1500^\circ C$ and 30,000 atmospheres to $-Al_2O_3$ plus liquid. From the P-T data the H for this reaction was calculated to be 18.5 kcal/mol. The reaction begins at the grain boundaries and proceeds into the kyanite grains. It was found that the microstructure of kyanite can be revealed by using H_3PO_4 as an etchant. When kyanite is "Hot-Pressed" in its stability region, each grain shows a complex slip and kink pattern; but when kyanite is formed under the same P-T conditions from the other polymorphs, and alusite or sillimanite, the microstructural features in each grain are more simple and are thought to be due to twinning under a more uniform stress configuration. A region of liquid immiscibility which might be expected at high pressures and temperatures in this system was not found.

October 1960

SUBJECT: INFLUENCE OF ENVIRONMENT ON CERAMIC PROPERTIES

INVESTIGATOR: George R. Pulliam, Bruce G. Leonard

CONTRACT: AF 33(616)-5875, Douglas Aircraft Company, Inc.

ABSTRACT: This study was concerned with environmental and non-stoichiometric effects on three different materials. The materials used were ZrO_2 , $AgCl$, and MgO single crystals. Definite evidences of low temperature plastic strain were found for O-deficient ZrO_2 . These evidences were non-linear stress-deflection curves at room temperature, appearance of slip lines on large grains after stress, and creep of ZrO_2 at $350^\circ C$. The creep of polycrystalline $AgCl$ was found to be highly sensitive to electromagnetic radiation. When an $AgCl$ specimen in creep was illuminated with $440\mu\text{m}$ light the creep rate was reduced. Light of $700\mu\text{m}$ caused the previously reduced creep rate to be increased. It is felt

WADD TR 60-338 (Continued)

that precipitates of photolytic silver hardened the AgCl in much the same manner as the precipitates in the precipitation hardenable stainless steels. The 700 μ m light had the effect of bleaching the precipitates out, and thus, permit the creep to continue. Creep specimens illuminated with 700 μ m light with no previous 440 μ m treatment were unaffected by the irradiation. MgO single crystals colored brown by u.v. irradiation were found to have an absorption peak at 4.3ev associated with oxygen. Ultra-violet irradiation increased the height of this peak so that it tailed off in the visible region. It is this tailing off that imparted the brown color to the crystals. Vacuum treatment of such specimens to 1300°C removed the peak and prevented further coloration by ultra-violet illumination.

WADD TR 60-377, OTS Release

September 1960

SUBJECT: STABILITY OF CERAMIC MATERIALS AT TEMPERATURES TO 2000°C
INVESTIGATOR: Joan Berkowitz
CONTRACT: AF 33(616)-6154, Arthur D. Little, Inc.
ABSTRACT: A study was made of the oxidation resistance of a number of commercial samples of MoSi₂ and WSi₂ in the temperature range 1600-1700°C. A thermal conductivity technique was developed to measure oxygen pick-up by the samples continuously. From the thermal conductivity measurement and the net weight change of the samples, an estimate was made of the rates of formation of volatile and non-volatile oxides. On the basis of X-ray analysis, oxidation products are believed to be volatile XO₃ (vapor) and non-volatile glass of the form SiO₂. nXO_m where X = Mo or W; n and m are not known. Tests were conducted at a total pressure of one atmosphere in a helium stream containing about 10 mm of oxygen and flowing at 119 cc/min. The oxidation of WSi₂ was also studied in a helium stream containing 5 mm of water vapor. The principal solid product in this case was metallic tungsten.

WADD TR 60-473, OTS Release

August 1960

SUBJECT: SURFACE AND ENVIRONMENTAL EFFECTS ON CERAMIC MATERIALS
INVESTIGATOR: P. Gibbs, G. S. Baker, B. Gale Dick, Jr., E. K. Beauchamp,
M. H. Miles, W. G. Rogers
CONTRACT: AF 33(616)-5876, University of Utah
ABSTRACT: Creep and recovery phenomena in sintered polycrystalline alumina have been studied. Effects of temperature and impurity content on creep behavior and the results of observation of polished and etched surfaces are discussed. The results are analyzed in terms of a tentative conventional mechanical analog.

An electrical study of the behavior of silver ions on a corundum surface is described. Current flow between evaporated silver films

WADD TR 60-473 (Continued)

on Al_2O_3 crystals is investigated as a function of geometry, temperature, atmosphere, history, and applied voltage. Migration of silver in some cases leaves visible deposits on the uncoated crystal. In general, the phenomena are complex and atmosphere-dependent.

The initial stages of creep in single crystals of α Al_2O_3 (synthetic sapphire) form of 0.1 inch diameter cylinders three inches long were loaded in three point loading to give slip on the (0001) $\langle 11\bar{2}0 \rangle$ slip system. Creep rates were measured in the temperature range 1000°C to 1200°C and in the stress interval of 500 to 1000 Kg per cm^2 resolved shear stress on the outermost fiber of the specimen. The stresses covered the region from too little stress to initiate creep up to the fracture stress. An approximate sixth power dependence of creep rate on stress was observed. The dependence of creep rate on temperature was measured for total creep less than 1% strain. A plot of log creep rate versus reciprocal temperature gives an activation energy of 85 Kcal/mole. Specimens oriented so that (1120) $\langle 1100 \rangle$ slip system was operative but the (001) $\langle 1120 \rangle$ slip system had zero resolved shear stress gave no significant creep at these temperatures.

WADD TR 60-749, Part I, OTS Release

February 1961

SUBJECT: FACTORS AFFECTING THERMAL SHOCK RESISTANCE OF POLYPHASE CERAMIC BODIES

INVESTIGATOR: P. T. B. Shaffer, D. P. H. Hasselman, A. Z. Chaberski

CONTRACT: AF 33(616)-6806, The Carborundum Company

ABSTRACT: An investigation of the factors which effect the thermal shock resistance of polyphase ceramic systems has been conducted using the model system zirconium carbide-graphite. The research has been divided into two areas:

1. Theoretical calculations of the individual material properties, which show that the included graphite particles may be regarded as spherical pores.

2. Experimental data, which substantiate the postulate that increased thermal shock resistance is a result of an increase in the ratio of strength over Young's modulus, brought about by the addition of the low Young's modulus phase.

WADD TR 60-782, Part I, OTS Release

November 1960

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE PART I. Mass Spectrometric Determination of the Dissociation Energies of the Molecules AgAu , AgCu , and AuCu

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WADD TR 60-782, Part I (Continued)

INVESTIGATOR: Marcel Ackerman, Fred E. Stafford, J. Drowart
CONTRACT: AF 61(052)-225, University of Brussels
ABSTRACT: The vapors issuing from mullite and Graphite Knudsen cells containing pure metals and alloys of the triad Cu-Ag-Au have been analyzed mass spectrometrically. From the experimental ratios of diatomic to monoatomic species and the vapor pressures of the elements, the following dissociation energies are obtained:

$$\begin{array}{ll} D_0^\circ (Cu_2) = 43.2 \pm 2.2 \text{ kcal} & D_0^\circ (AgAu) = 47.6 \pm 2.2 \\ D_0^\circ (Ag_2) = 35.7 \pm 2.2 \text{ kcal} & D_0^\circ (AgCu) = 40.7 \pm 2.2 \\ D_0^\circ (Au_2) = 51.9 \pm 2.2 \text{ kcal} & D_0^\circ (AuCu) = 54.3 \pm 2.2 \end{array}$$

These are based on $H_{298}^{\text{vap}} = 81.1, 68.4$ and 87.5 cal/mole for Cu, Ag and Au where D_0 of AgAu depends on the value for Cu, and D_0 of AgAu and AuCu on Au. The uncertainties quoted do not include the uncertainty in ΔH^{vap} .

These results are interpreted in terms of chemical bonding theories. The relation between bonding in the gas and in the pure condensed phases is again observed. Furthermore, a previously unobserved qualitative relation between the dissociation energy of the asymmetrical molecule and the heat of formation of the corresponding alloys is indicated.

WADD TR 60-782, Part II, OTS Release

November 1960

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE PART II. Mass Spectrometric Studies of the Vaporization of Sulphides and the Dissociation Energy of S_2
INVESTIGATOR: R. Colin, P. Goldfinger, M. Jeunehomme
CONTRACT: AF 61(052)-225, University of Brussels
ABSTRACT: The equilibria $S_2 \rightleftharpoons 2S$ and $CaS \rightleftharpoons Ca + S$ have been observed in the vapor phase above CaS. $D_0(S_2) = 4.4 \pm 0.2 \text{ e.v.}$, $D_0(SO) = 5.4 \pm 0.2 \text{ e.v.}$, $D_0(CaS) = 3.7 \pm 0.2 \text{ e.v.}$, and $\Delta H_{298}^{\text{vap}}(CaS) = 5.9 \pm 0.2 \text{ e.v.}$ have been determined mass spectrometrically.

WADD TR 60-825, OTS Release

March 1961

SUBJECT: REFRACTORY COATINGS FOR TUNGSTEN
INVESTIGATOR: C. G. Goetzel, P. Landler
CONTRACT: AF 33(616)-6868, New York University
ABSTRACT: A preliminary study of the feasibility of protecting tungsten wires against oxidation at 3300°F (1815°C) was undertaken. A tungsten disilicide coat was found to be the most promising coating for this operating temperature.

WADD TR 60-825 (Continued)

Several siliconizing methods were given a cursory treatment, including vapor plating and dipping into molten copper-silicon bath, and the simplest, cementation, selected for closer investigation. This operation was performed under flowing hydrogen atmosphere and such variables as temperature, time, and composition of the cementation pack were studied as a main part of our exploratory work. A single brittle tungsten-disilicide (WSi_2) layer is formed by this method. The inherent brittleness of tungsten appears further aggravated by the development of micro-cracks in the layer as well as in the substrate during siliconizing.

Oxidation tests using direct electrical resistance heating were performed. It was found that pack-siliconized wires can withstand oxidation at $3300^{\circ}F$ in still air for prolonged periods up to and exceeding 10 hours. This is accomplished without any destruction of the coating in the high temperature zone owing to the formation of a viscous glassfilm on the surface. A pronounced tendency toward self-healing is a useful property of this type of coat.

A serious drawback of the disilicide coat, however, is its low temperature oxidation ("Disilicide Pest") which occurs in parts exposed to temperatures between 1200° and $2280^{\circ}F$ (650° - $1250^{\circ}C$) for prolonged periods. It causes a destruction of the disilicide layer over a period of 5 to 15 hrs, depending on coating thickness. It was the actual life determining factor in those tests where other failures caused by improper technique had been eliminated.

Preliminary attempts to deposit ZrO_2 on top of siliconized wires by means of poor adhesion. Where bonding was obtained, no improvement in life under high temperature oxidizing conditions could be ascertained.

GRAPHITE

WADC TR 58-360, Part III, OTS Release

February 1961

SUBJECT: INVESTIGATION OF ELASTIC AND THERMAL PROPERTIES OF CARBON-BASE BODIES
INVESTIGATOR: S. Mrozowski, J. F. Andrew, N. Juul, H. E. Strauss, D. C. Wobischall
CONTRACT: AF 33(616)-5186, University of Buffalo
ABSTRACT: In continuation of the work the elastic properties and permanent set were investigated at room temperature for carbons made of graphitized filler (soft and hard) and for carbons impregnated in the baked or graphitized state in dependence on the heat-treatment temperature. An apparatus for study of deformation at high temperatures (1000 - $3000^{\circ}C$) was built and calibrated and the first results for the variation of Young's modulus with

WADC TR 58-360, Part III (Continued)

ambient temperature were reported. Another greatly improved apparatus for studies of heat conductivity at high temperatures was finished and also put into operation. Further studies of corrections to the heat conductivity due to convection by gas across the pores were made and it is shown that such corrections are necessary even in the case of quite dense carbons. Transverse heat diffusivity was investigated in the temperature range 700 - 2800°C by a transient state method, using a newly constructed apparatus; good fit with the previously found data in the low temperature range is obtained. A general discussion of the aims of the project concludes the report.

WADC TR 59-789

April 1960

SUBJECT: DEVELOPMENT OF GRAPHITE AND GRAPHITE BASE MULTI-COMPONENT MATERIALS FOR HIGH TEMPERATURE SERVICE
INVESTIGATOR: W. W. Lozier
CONTRACT: AF 33(616)-5563, National Carbon Company
ABSTRACT: This report describes graphite-type materials and their evaluation in various test facilities. Arc torches of various characteristics were employed.

Materials found promising in their oxidation resistance in previous contract work were confirmed and subjected to more severe tests with higher sample temperature and greater heat flux. At sample temperatures of 3000 K or above, none of the variations showed ablation resistance much better than unmodified graphite itself.

The analysis of the ablation data on plain graphite has suggested a marked dependence of the heat of ablation on the "enthalpy potential" and resulted in heats of ablation as high as 16000 B.t.u./lb.

One material which has been found to show unique behavior is a highly oriented graphite deposited by pyrolytic decomposition of a hydrocarbon gas. These pyrolytic graphites have been made and tested in various shapes and sizes. Because of their highly anisotropic conductivity, the pyrolytic coatings have exhibited a marked ability to retard heat penetration into and temperature rise of the substrate material. However, the pyrolytic graphite has shown no conclusive advantage in ablation resistance under the conditions of the tests. Seeming advantages in some cases may turn out to be due to indirect effects such as a different distribution among various heat dissipation mechanisms.

A new method developed for the measurement of reflectance and emissivity using the carbon arc image furnace has given new information on the spectral emissivity of graphite and lampblack at temperatures ranging from 500°K to 3800°K. It has also permitted a more accurate determination of the temperature of the "pyrometric" carbon arc which is widely recognized and used as a high temperature standard.

HIGH STRENGTH METALS

WADC TR 58-478, Part II, OTS Release

September 1960

SUBJECT: DEVELOPMENT OF WROUGHT BERYLLIUM ALLOYS OF IMPROVED PROPERTIES
INVESTIGATOR: John G. Klein, Leslie M. Perelman, Wallace W. Beaver
CONTRACT: D.O. 33(616)-57-19, The Brush Beryllium Company
ABSTRACT: Mechanical and physical properties are reported for extruded and/or rolled products fabricated from beryllium-rich alloys of silver, tin, cadmium, zinc, nickel, and copper, as well as beryllium fabricated from subsieve-size powder and powder of higher than normal beryllium oxide content.

WADC TR 59-63, Part II, OTS Release

July 1960

SUBJECT: DEVELOPMENT OF LOW ALLOY STEEL COMPOSITIONS SUITABLE FOR HIGH STRENGTH STEEL CASTINGS
INVESTIGATOR: Hugo R. Larson, Ronald C. Campbell, Herbert W. Lloyd
CONTRACT: AF 33(616)-6485, American Brake Shoe Company
ABSTRACT: Cast high strength steels were investigated in an effort to facilitate the production of high integrity, high strength castings for aircraft and missile applications.

A study was made of heat treatments and compositions for optimum properties in heavy sections. Acceptable properties were developed in the modified 8735 and 4335 alloys in two and three inch sections, but the problem of poor ductility in heavy sections of 5% chromium steel was not solved.

Spectrographic analyses and sulfur additions to high purity heats failed to reveal the factors responsible for improved properties in high purity heats. Fatigue properties of normal and high purity and vacuum melted low alloy steels were rather disappointing. An endurance limit of approximately 55,000-60,000 psi was observed.

Excellent properties were developed at room temperature and 1000°F in the 5% Cr air hardening steel with a 3% tungsten addition and proper heat treatment. The effect of soundness and feeding distance on the properties of the 5% Cr air hardening steel was also studied.

Finally, analysis of production heats indicated good reproducibility of properties in cast high strength steels. High purity production heats were shown to be somewhat superior to normal purity heats.

WADC TN 59-326, OTS Release

July 1960

SUBJECT: THE STATUS OF RESEARCH AND DEVELOPMENT FOR HIGH STRENGTH AIRCRAFT STEELS

WADC TN 59-326 (Continued)

INVESTIGATOR: E. M. Kennedy, Jr.
ABSTRACT: New aircraft and missile developments have required the development and use of structural steels having optimum strength and ductility over a wide range of temperatures.

In this survey an attempt is made to point up to some of the significant areas of investigation in which efforts are being expended toward developing and improving steels having tensile strengths in excess of 260,000 psi. In order to complete the survey of the field of interest, the sources known to have research and development interest in this area were contacted either by personal visitation or through official correspondence.

The results of the contacts made were gratifying and the many investigations and studies being carried on are commented upon in the text of this report.

WADD TN 59-424, Part II, OTS Release

January 1961

SUBJECT: PRELIMINARY ANALYSIS OF THE CAPABILITIES OF A COMPOSITE SLAB FOR AN ADVANCED HEAT-SINK DESIGN
INVESTIGATOR: Paul A. Libby
CONTRACT: AF 33(616)-5944, Polytechnic Institute of Brooklyn
ABSTRACT: Presented here are the results of a numerical analysis of the capability of a composite slab of beryllium oxide (BeO) and beryllium (Be) to absorb the heating associated with the reentry of a high performance ballistic missile. The trajectory considered corresponds to a ballistic factor ($W/C_D A$) equal to 2,000 psf, to a reentry velocity of 20,000 fps, and to a reentry angle of 20° . The thermal properties of the metals were considered temperature dependent; surface radiation was included. An implicit system of numerical integration was applied.

The numerical results indicate that the maximum permissible heat transfer rates for the trajectory are obtained with a relatively thin slab of beryllium oxide. The addition of beryllium to this slab may be required for structural and thermal shock considerations but does not greatly improve the heat-sink capabilities.

The permissible values of the heat transfer parameter are applied to a slender cone with a 20° half angle and with a spherical cap of 0.25 ft nose radius. The laminar and turbulent heat transfer data for such a cone were available. It is shown that for laminar flow no heat transfer reduction is required on the cone while for turbulent flow a reduction to $1/2$ is required. The possibility of utilizing a mass transfer system in order to reduce the heat transfer on the spherical cap and on the cone in the case of turbulent flow is briefly discussed.

SUBJECT: EVALUATION AND ALLOY DEVELOPMENT OF HOT-WORK DIE STEELS FOR STRUCTURAL PURPOSES

INVESTIGATOR: Michael Schneider, John P. Sheehan

CONTRACT: AF 33(616)-5633, Armour Research Foundation

ABSTRACT: A program to investigate the tensile properties of three commercial hot-work die steels, *Vascojet* 1000, Potomac M, and Peerless 56 has been carried out. The results indicate that in sheet form these materials are essentially equivalent and develop the following nominal peak properties when air cooled from 1850°F and double tempered at 1000°F: 295,000 psi ultimate tensile strength; 233,000 psi yield strength at 0.2% offset; and 7% elongation in 2 inch gage length. Those materials which had been cross rolled showed little or no difference between longitudinal and transverse properties. Furthermore, no significant differences were obtained in longitudinal tensile properties by vacuum arc remelting of commercial bar stock.

Approximately 30 experimental alloy steels were melted, in which most of the common alloying elements were investigated to determine their effects on tensile properties. Of these, several developed yield strengths of 280,000-290,000 psi, ultimate tensile strengths of 340,000-360,000 psi and tensile elongations of 3-5% in 2 inch gage length on sheet material approximately 0.080 inch thick.

SUBJECT: FURTHER STUDIES ON ACTIVE-EUTECTOID ALLOYS ON TITANIUM

INVESTIGATOR: R. F. Bunshah, D. Osterberg, E. Ence, H. Margolin

CONTRACT: AF 33(616)-5655, New York University

ABSTRACT: This report covers further work succeeding on previous investigations on active eutectoid titanium-copper alloys in which decomposition of the beta phase to alpha plus compound occurs rapidly. The current program is divided into two parts. Part I deals with the properties of active eutectoid alloys. The effect of stepwise additions of Al and/or Zr to binary Ti-5Cu, Ti-6Cu, Ti-6Ni, Ti-2Ni-2Cu and Ti-4Ni-2Cu alloys on the tensile strength at room and elevated temperatures, creep-rupture properties and thermal stability under stress were studied. Several alloys exhibited outstanding short time tensile strengths up to 1200°F much superior to the best competition. The creep rupture properties were correspondingly good. These alloys which exhibited highest strengths also showed instability. Stability could be restored partially by some sacrifice in strength.

This and the previous work has demonstrated that a new class of titanium alloys, i.e., of the alpha-compound type have been developed. They have potential utility in applications where short time elevated temperature strength properties are the prime consideration particularly for one cycle operation. Part II is concerned with the role played by Zr in strengthening these alloys. Constitutional diagram studies on the Ti-Zr and the 750°C section of the Ti-Cu-Zr systems were undertaken.

WADD TR 60-99

May 1960

SUBJECT: INVESTIGATION OF THE Ti-Al-Cb SYSTEM AS A SOURCE OF ALLOYS FOR USE AT 1200° - 1800°F
INVESTIGATOR: Joseph B. McAndrew, Charles R. Simcoe
CONTRACT: AF 33(616)-6125, Armour Research Foundation
ABSTRACT: The work reported was an experimental study of titanium base alloys containing major additions of both aluminum and columbium, with attention primarily directed toward the properties of such alloys in the temperature range of 1200°F to 1800°F.

Alloys were prepared with aluminum content of 5 to 17.5%, plus 15 to 30% columbium, and were investigated with respect to forgeability, density, oxidation resistance at 1000°C (1832°F), hardness, short-time tensile properties at room temperature and elevated temperatures, heat treatment response, and microstructure. Several alloys were also subjected to bend, impact and stress-rupture tests. The refinement, precision, or extent of testing was not such as would produce data appropriate for design purposes, since the program was intended to yield findings suitable for research uses.

A considerable range of properties was exhibited by alloys of various compositions. Several alloys of high aluminum content had exceptionally high strength-to-density ratios in short-time tensile tests at temperatures up to 1600°F (871°C). Forgeability, good oxidation resistance, and low density were attractive features shared by all of the alloys. Many of them showed considerable response to heat treatment.

Analysis of the data indicates that the Ti-Al-Cb alloys offer a very promising field in which to conduct further research.

WADD TR 60-116, OTS Release

July 1960

SUBJECT: BERYLLIUM CRACK PROPAGATION AND EFFECTS OF SURFACE CONDITION
INVESTIGATOR: C. O. Matthews, M. I. Jacobson, W. E. Jahsman, W. V. Ward
CONTRACT: AF 33(616)-5978, Lockheed Missiles and Space Division,
California Division of Lockheed Aircraft Corporation
ABSTRACT: The mechanical properties of beryllium sheet with various surface finishes were investigated. The best properties were obtained on sheet which had been etched to remove surface defects caused by machining.

Ductility was low in all cases, being limited by the presence of notches and by preferred orientation in the sheet. Ductility was increased by heating to 400°F to 600°F.

The fatigue endurance limit was as high as the static tensile strength, and was improved by etching. Impact tests were found to be most suitable for distinguishing between various surfaces.

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Theoretical analyses were made of the effect of including residual stress in the Griffith crack theory, and of the dynamic stress at the leading edge of a crack in a uniaxially stressed plate.

WADD TR 60-120, OTS Release

June 1960

SUBJECT: STUDY OF FATIGUE PROPERTIES OF ULTRA-HIGH STRENGTH STEEL
INVESTIGATOR: Harvey B. Nudelman, John P. Sheehan
CONTRACT: AF 33(616)-6290, Armour Research Foundation
ABSTRACT: A temper-resistant, high-strength steel was investigated with reference to fatigue strength. This alloy was prepared by two different melting techniques. The first method consisted of induction melting in air using standard deoxidation techniques. The second method involved the application of a special deoxidation practice to induction melting; silicon-free steel was deoxidized with carbon and aluminum to minimize the presence of silicate inclusions. These alloys were tested in fatigue using the Prot accelerated method. The results of the samples melted with standard practice indicated that fatigue strength is improved slightly by tempering at elevated temperature. The application of a special melting process showed that a very significant increase in the Prot fracture stress to ultimate tensile strength ratio (E_p/UTS) can be obtained by eliminating the presence of silicon and de-oxidizing with carbon and aluminum.

WADD TR 60-132, OTS Release

October 1960

SUBJECT: REFRACTORY METAL CONSTITUTION DIAGRAMS
INVESTIGATOR: A. R. Kaufmann, E. J. Rapperport, M. F. Smith, J. Wulff, J. Brophy, N. J. Grant, B. C. Giessen, A. Taylor, N. Doyle
CONTRACT: AF 33(616)-6023, Nuclear Metals, Inc., Massachusetts Institute of Technology, Westinghouse Research Laboratories
ABSTRACT: Nine binary constitution diagrams and one ternary constitution diagram of some of the refractory metals are presented. The binary diagrams include Hf-Mo, Hf-Re, Hf-W, Nb-Re, Os-Ta, Os-W, Re-Ta, Ru-Ta, Ru-W, and the ternary is Re-Ta-W.

Care was taken to obtain reliable diagrams. In particular, the purity of the constituents (99.9 percent plus) was protected at all times, and the temperatures were measured to an accuracy within $\pm 20^\circ\text{C}$.

WADD TR 60-258, OTS Release

June 1960

SUBJECT: A STUDY OF THE TITANIUM-LIQUID OXYGEN PYROPHORIC REACTION
INVESTIGATOR: J. D. Jackson, P. D. Miller, W. K. Boyd, F. W. Fink
CONTRACT: AF 33(616)-6345, Battelle Memorial Institute

WADD TR 60-258 (Continued)

ABSTRACT: A review of the literature indicates that titanium is impact sensitive under liquid oxygen below the acceptable limit for other metals.

An experimental program was begun to determine the mechanism of the titanium-LOX reaction. Several factors were investigated singly in a controlled manner using unalloyed titanium (75A) and an alloy (6Al-4V) that were carefully cleaned. The factors were:

- (1) Exposure of a fresh surface by fracture and tearing
- (2) Deformation by impact using steel balls
- (3) Impact of smooth specially cleaned flat surfaces
- (4) LOX pressure and velocity
- (5) Galling

The results from this program indicate that no one of the above, per se, is a primary cause of this reaction. A proposed mechanism is that heat generated by impact produces gaseous oxygen, which is compressed at local sites. A fresh surface exposed by the impact reacts with the high-pressure gaseous oxygen. Propagation is dependent on the amount of heat generated and the rate of heat loss from the affected area.

WADD TR 60-316, OTS Release

August 1960

SUBJECT: BINARY AND TERNARY DIAGRAMS
INVESTIGATOR: Elmars Ence, Paul A. Farrar, Harold Margolin
CONTRACT: AF 33(616)-5704, New York University
ABSTRACT: I. Ti-Al-Cr System - The Ti-rich corner of the Ti-Al-Cr system has been reinvestigated from 600 - 1400°C (up to 40 wt percent Al and 32 wt percent Cr). Partial isothermal sections have been constructed at 1400, 1200, 1100, 1000, 800 and 600°C. The phases encountered in the portion of the system investigated are α , β , γ_{Ti_3Al} , δ_{Ti_2Al} , ϵ_{TiAl} and $TiCr_2$. A miscibility gap in the β field is produced by the interaction of the β + δ_{Ti_2Al} and the β + $TiCr_2$ fields.

II. Ti-Al-V System - The Ti-Al-V system has been reinvestigated from 50 to 100 wt percent titanium and from 500°C to 1400°C, using X-ray diffraction and metallographic techniques. Isothermal sections were delineated at 500, 600, 700, 800, 900, 1000, 1100, 1200, 1300 and 1400°C. Vertical sections at 2, 4, 6, 10, and 16 wt percent aluminum; 2, 4, 6, 10, and 16 wt percent vanadium and 90 wt percent Ti were constructed. The phases encountered in the portion of the system reinvestigated were α , β , γ_{Ti_3Al} , δ_{Ti_2Al} and ϵ_{TiAl} .

WADD TR 60-340, OTS Release

September 1960

SUBJECT: VOID FORMATION AND GRAIN BOUNDARY SLIDING IN ALUMINUM-MAGNESIUM SOLID SOLUTION ALLOYS
INVESTIGATOR: Arthur W. Mullendore, Nicholas J. Grant
CONTRACT: AF 33(616)-5926, Massachusetts Institute of Technology
ABSTRACT: Void formation and growth during creep were observed in aluminum - 1.92 percent magnesium and aluminum - 5.10 percent magnesium as a function of temperature, stress, and time. The origin of voids is related to the development of an irregular (serrated) grain boundary and to grain boundary sliding. Grain boundary sliding is shown to result largely from crystal slip crossing the grain boundary.

WADD TR 60-425, OTS Release

September 1960

SUBJECT: MECHANICAL PROPERTIES OF BERYLLIUM
INVESTIGATOR: A. E. Riesen, R. T. Ault
ABSTRACT: The test procedures and results of a mechanical properties' determination program which included tensile, torsion, pin shear, creep rupture, and fatigue data on two lots of beryllium are presented. One lot of material was hot pressed with a BeO content of 1.45%; the other lot was hot pressed and hot extruded and contained 1.55% BeO.

Emphasis was placed on the fatigue and creep rupture data. Fatigue tests were conducted at room and elevated temperatures with stress ratios of $A = \infty$ and 0.67. Both lots of material showed surprising strength under fatigue loading conditions whereas beryllium's inherent brittleness manifested itself under static loading conditions by the brittle fractures.

The hot pressed-hot extruded material was uniformly stronger and therefore more desirable for design purposes than the material that was only hot pressed.

WADD TR 60-443, OTS Release

September 1960

SUBJECT: PARTITIONING AND MICRODISTRIBUTION OF INTERSTITIAL ALLOYING IN TITANIUM
INVESTIGATOR: Irving B. Cadoff, Joseph Winter
CONTRACT: AF 33(616)-5506, New York University
ABSTRACT: The partitioning or segregation of excess interstitial solutes at the grain boundaries in alpha phase and beta phase, titanium alloys was investigated. Internal friction spectra of dilute alloys of titanium with oxygen, nitrogen and carbon were obtained. From these spectra the activation energy for grain boundary stress relaxation, the net interaction energy of a solute atom with a grain boundary, and the excess solute concentration were calculated.

As in previous investigations of this type the partitioning of excess solute was found to obey a relation of the form:

WADD TR 60-443 (Continued)

$$C_s = C_0 \exp^{-\frac{V}{s/kT}}$$

The interaction energies were found to lie in the range 0.15 to 0.40 eV for binary interstitial alloys. The expected behavior of increase in activation energy, decrease in peak height and decrease in peak temperature with increasing solute concentration was observed. In the case of carbon and nitrogen alloys the activation energy increase exhibited a saturation effect. While this was not observed in oxygen alloys, the nature of the curve indicated approach to saturation. In the range of saturation, the carbon alloys developed a second peak, the composition at which the peak was fully evident being close to the solubility limit of carbon in alpha titanium. From the nature of the curves obtained it may be inferred that the double peak may be associated with the formation of the delta phase, which causes local depletion of the carbon atoms segregated at the grain boundary.

Tensile measurements indicated that the increase of excess solute at grain boundaries and dislocations brought about by either increasing the bulk concentration or decreasing the equilibrium annealing temperature resulted in an increase in yield strength. The effect of excess solute on yield stress was found to be parallel to its effect on the internal friction spectra.

WADD TR 60-542, OTS Release

September 1960

SUBJECT: STUDY OF RATE CONTROLLING PROCESS FOR COMPRESSIVE DEFORMATION OF HIGH PURITY ALUMINUM

INVESTIGATOR: A. E. Riesen

ABSTRACT: A unique testing technique for determining the rate-controlling process for compressive deformation of high purity aluminum over the temperature range of 297°K to 358°K is discussed, and an attempt is made to correlate the activation energy for compression with some recognized rate-controlling process for tensile deformation of aluminum. It is shown that the compressive activation energy obtained, 5,400 cal/mol., cannot be related to any known tensile deformation process.

The results of this study indicate a need for further experimental investigations to define the compression activation energy spectrum for aluminum over a broad temperature range.

HIGH TEMPERATURE METALS

WADC TR 59-19

April 1960

SUBJECT: DEVELOPMENT AND EVALUATION OF HIGH-TEMPERATURE TUNGSTEN ALLOYS

INVESTIGATOR: F. C. Holtz, R. J. Van Thyne

CONTRACT: AF 33(616)-5218, Armour Research Foundation

ABSTRACT: Alloys containing a minimum of 90 wt% tungsten were developed for use at temperatures up to 2000°F. Compositions based on the W-Ni-Fe system were prepared by cold-pressing blended metal powders, followed by liquid-phase sintering in hydrogen. The sintered products consisted of rounded, tungsten-rich grains in a W-Ni-Fe solid solution matrix. Ternary alloys containing up to 98 wt% tungsten were investigated; materials at the 95 wt% tungsten level were very ductile and could be rolled at room temperature. The hardness of cold-rolled specimens decreased upon annealing at temperatures above 1400°F. Maximum tensile strength values of 34,000 psi at 2000°F occurred at the 97 wt% tungsten level. Tensile elongations decreased above 1000°F, and these ternary materials had a 100-hour rupture life at a stress level of about 2500 psi at 2000°F.

The effects of tungsten contents, nickel-to-iron ratios, and quaternary additions on properties of W-Ni-Fe alloys were studied at room temperature and at 2000°F. Increasing amounts of tungsten resulted in lower ductility and slightly higher strength; a slight improvement in stress-rupture life at 2000°F was noted. Optimum nickel-to-iron ratios were dependent on the tungsten levels. Proportionally higher iron contents increased tensile strength and decreased elongation at elevated temperature. The elements Al, Cr, Mo, Nb, Ta, and Ti were added to a 90W-Ni-Fe-base. Room temperature strength and ductility of this base were lowered by 2.5 wt% of Cr, Nb, and Ta, and by 0.5 wt% of Al and Ti. Moderate improvements in elevated temperature properties were noted in alloys containing 2.5 wt% Cr, Mo, and Ta. Oxidation resistance, slightly inferior to that of unalloyed tungsten, was not materially affected by the small quaternary additions.

Blended W-Ni-Fe powders were hot-pressed in graphite dies, using induction heating. The compacts were relatively fine-grained and had a thin carbide layer on the surfaces. Densities were usually above theoretical values due to squeeze-out of the very fluid matrix phase. Ingots of unalloyed tungsten and a 90W-10Nb alloy prepared by consumable-electrode arc-casting had a coarse, columnar grain structure and were not amenable to working.

WADC TR 59-29, Part II, OTS Release

July 1960

SUBJECT: AN INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR VERY HIGH TEMPERATURE APPLICATIONS

INVESTIGATOR: Robert M. Paine, A. James Stonehouse, Wallace W. Beaver

CONTRACT: AF 33(616)-56-12, Brush Beryllium Company

WADC TR 53-373, Sup 8

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WADC TR 59-29, Part II (Continued)

ABSTRACT: The preparation, fabrication, and properties of intermetallic compounds under development for service in the temperature range of 2300° to 3000°F are described. The compounds, principally beryllides, have exhibited strengths as measured by the modulus-of-rupture test of up to 40,000 psi at 2700°F and resistance to oxidation for 100 hours to 2800°F and have indicated good thermal conductivity.

The compounds were prepared by solid-state reactions and fabricated chiefly by hot-pressing techniques. Oxidation tests were made in dry air at 2500° to 3000°F and in moist air (57°F dew point) at 2300° to 2500°F. Transverse-rupture tests were carried out at 2300°, 2500°, and 2750°F. Room temperature hardness data and melting points are reported for some compounds. Thermal-expansion and thermal-conductivity data for selected compounds are also presented.

The intermetallic compounds included in the investigations were NbAl₃, TaAl₃, CrBe₂, MoBe₁₂, Nb₂Be₁₇, NbBe₁₂, TaBe₂, Ta₂Be₁₇, TaBe₁₂, TiBe₂, TiBe₁₂, ZrBe₁₃, Cr₃Si, Ti₅Si₃, TiSi, and TiSi₂, with CrBe₂, TaBe₂, TiBe₂ and the silicides receiving only a very limited effort (chiefly oxidation tests). The more promising intermetallic compounds for high-temperature applications are concluded to be ZrBe₁₃, Nb₂Be₁₇, NbBe₁₂, Ta₂Be₁₇, and TaBe₁₂.

WADD TR 60-37, OTS Release

May 1960

SUBJECT: PHYSICAL METALLURGY OF TUNGSTEN AND TUNGSTEN BASE ALLOYS
INVESTIGATOR: Russell H. Atkinson
CONTRACT: AF 33(616)-5632, Westinghouse Lamp Division
ABSTRACT: To establish a comparative standard for evaluation of changes in properties brought about by the addition of alloying constituents to tungsten, base line data and fundamental physical metallurgical information on tungsten of various purity levels were obtained and studied.

Base line data on high purity and commercial powder metallurgy tungsten have revealed differences in tensile transition and recrystallization temperatures believed attributable to variations in metallic impurity concentrations. High temperature (to 2700°F) tensile properties of these materials are little affected by differences in levels of trace impurities except for the possibility that observed anomalous and poorly reproducible creep rupture behavior is impurity-induced.

The tensile transition temperature of electron beam zone melted single crystal tungsten lies between -196° and -107°C, the main influence being surface condition. Deformation of these crystals is accompanied by twinning, and observed low temperature yield point phenomena are tentatively attributed to hydrogen.

Polycrystalline tungsten exhibits several low magnitude internal friction peaks of undetermined origin in the 20°-800°C region.

WADD TR 60-37 (Continued)

Annealed single crystals show no such behavior.

Purposeful introduction of carbon by carbiding and annealing and of oxygen by either direct gas-metal reaction or soaking in tungstic oxide at elevated temperatures does not produce homogeneous distribution of these elements in concentrations above 20-40 weight parts per million.

Addition of thoria as a dispersed second phase to tungsten raises the recrystallization temperature of tungsten and thus improves the high temperature (2700°F) yield strength by a factor of 2-3.5 and the 100 hour creep rupture strength by a factor of about 2. Significant improvement over these values is indicated for the addition of tantalum carbide in place of the thoria. Other dispersed second phase systems employing ZrO₂, B₄C, NbC, SiO₂, or WB have been studied. Data are incomplete.

WADD TR 60-155, OTS Release

June 1960

SUBJECT: DEVELOPMENT OF METHODS AND INSTRUMENTS FOR MECHANICAL EVALUATION OF REFRACTORY MATERIALS AT VERY HIGH TEMPERATURES

INVESTIGATOR: D. H. Fisher, D. N. Gideon, G. M. McClure, H. J. Grover, R. L. Carlson, G. K. Manning

CONTRACT: AF 33(616)-6155, Battelle Memorial Institute

ABSTRACT: A mechanical-testing system has been established which is capable of providing tensile and compressive stress-strain data and shear-strength data up to 4000°F in vacuum. The results of an evaluation of the system using a molybdenum-0.5 per cent titanium alloy in the bar form are presented. Tension, compression, shear and bearing tests were conducted at temperatures up to 3500°F. A detailed description of the testing system and specimen designs is presented. An optical strain-measurement system which permits displacements to be measured directly in the gage section is described.

The state-of-the-art report on the development of equipment and techniques of mechanical-properties measurement is based on a survey of literature and discussions with researchers at Battelle and other laboratories. With the objective of developing equipment and techniques for use to 6000°F or higher, problems to be solved in order that accurate data will be obtained are considered. Recommendations are made concerning furnace design, temperature and strain measurement, and specimen, grip, and loading design. In particular, the probable advantages of graphite heaters coated with tantalum carbide are pointed out; plans for experimental investigations and a description of apparatus built to study carbide coatings are presented. The further investigation of two-color pyrometry was also recommended.

WADD TR 60-240, OTS Release

July 1960

SUBJECT: RESEARCH ON PROPERTIES OF HIGH STRENGTH MATERIALS SUITABLE FOR HIGH TEMPERATURE APPLICATIONS

WADC TR 53-373, Sup 8

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WADD TR 60-240 (Continued)

INVESTIGATOR: Harold N. Cummings, Foster B. Stulen, William C. Schulte
CONTRACT: AF 33(616)-6552, Curtiss-Wright Corporation

ABSTRACT: 1. Bars of iron-molybdenum alloys, intended for study as to suitability for ball bearings at temperatures up to 1000°F, were found to be so non-homogeneous and brittle that specimens could not be machined from them.

2. High temperature torsion tests of relaxation were made to study the suitability of two alloys, M-1 tool steel and Inconel X, for springs at 1000°F, and of two alloys, Waspalloy and Udiment 500, for springs at 1500°F. Alternating torsion of ~~10~~ ksi, superimposed on an initial steady stress of 25 ksi, was applied to specimens of each alloy. Inconel X lost about 10 to 15 percent of the steady load in about 100 hours. The three other alloys relaxed two to three times as much when tested at the respective temperature mentioned above.

WADD TR 60-343, OTS Release

August 1960

SUBJECT: INVESTIGATION OF DIFFUSION BARRIERS FOR REFRACTORY METALS
INVESTIGATOR: E. M. Passmore, J. E. Boyd, L. P. Neal, C. A. Andersson,
B. S. Lement

CONTRACT: AF 33(616)-6354, Manufacturing Laboratories, Inc.

ABSTRACT: Twenty-three base-barrier metal combinations were screened by annealing diffusion couples at 1700~~15~~°C. Measurements indicating the extent of interdiffusion between barrier and base metals were made by metallographic examination, microhardness tests, and electron microbeam analysis.

From these measurements, it is concluded that Hf and Ir are the most promising barriers for W at 1700°C, with V as a third choice. It appears to be the best choice for Ta and Mo. No recommendations could be made for Cb base metal, primarily because of melting at the screening temperature.

A comparison of interdiffusion behavior with base-barrier phase relations indicates no correlation with either solid solubility or presence of intermediate phases. The melting points of both barrier and base appear to be the most important factors affecting the extent of interdiffusion.

WADD TR 60-405, OTS Release

February 1961

SUBJECT: A STUDY ON CONTROLLING HIGH TEMPERATURE OXIDATION OF VANADIUM BASE ALLOYS

INVESTIGATOR: James D. Klicker, Howard B. Bomberger

CONTRACT: AF 33(616)-6113, Crucible Steel Company of America

ABSTRACT: The primary objective of this contract was to study means for inhibiting the rapid oxidation of vanadium alloys

WADD TR 60-405 (Continued)

The characteristics of 240 oxide mixtures were studied first to determine those elements whose oxides form refractory oxides with vanadium oxide. Melting points of these mixtures contained one or more of the following oxides: Al_2O_3 , Cb_2O_3 , CrO_3 , NiO_2 , TiO_2 , Y_2O_3 , ZrO_2 .

Twenty-eight of the more refractory oxides were then applied to vanadium sheet but they appeared to offer little protection as coatings.

Seventy laboratory-size alloy ingots were cast, processed, and tested for oxidation resistance. Vanadium alloys containing titanium, aluminum and nickel yielded the most refractory scales; and, oxide melting points as high as $1950^{\circ}F$ were observed. Some of the more promising compositions could be hot worked by rolling and extruding.

This study indicates that useful oxidation resistance may be possible through further alloy development.

WADD TR 60-566, OTS Release

December 1960

SUBJECT: SURFACE TENSION OF REFRACTORY METALS
INVESTIGATOR: S. Victor Radcliffe, H. Udin
CONTRACT: AF 33(616)-6269, Manufacturing Laboratories, Inc
ABSTRACT: An electron-bombardment heating technique has been applied to the measurement of the surface tension of solid niobium by a modification of the Udin method. The value obtained is 2100 dynes. cm^{-1} at $2250^{\circ}C$ in a vacuum of approximately 10^{-5} mm. This result is consistent with a theoretical estimate and provides support for Taylor's estimates of the surface tension of the refractory metals.

From measurements of the dihedral angle of grain boundaries formed in niobium by thermal etching, the interfacial energy (γ') was computed to be 0.36 of the surface energy.

An electrical-resistance heating technique has been applied to the measurement of the surface tension of solid tungsten, molybdenum and niobium by a modification of the Udin method. The method in this form was found to be unsuitable for these metals.

WADD TR 60-646, Part I, OTS Release

February 1961

SUBJECT: CARBONIZATION OF PLASTICS AND REFRACTORY MATERIALS RESEARCH
PART I
INVESTIGATOR: J. A. Coffman, G. M. Kibler, T. R. Riethof, A. A. Watts

WADC TR 53-373, Sup 8

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WADD TR 60-646, Part I (Continued)

CONTRACT: AF 33(616)-6841, General Electric Company

ABSTRACT: This report presents details of the conduct of, and the results obtained from, experimental investigations concerned with (1) the rate and mechanism of the carbonization of plastics; (2) the measurement of the vapor pressure of refractory materials; and (3) the determination of the spectral emissivities of refractory materials. These investigations are continuing.

Three complementary experimental techniques were, and are being used to study the carbonization of plastics. These are (1) thermogravimetric analysis, which gives an over-all picture of reaction rates under slow heating; (2) fluid-bed carbonization of powdered resin, which provides insight into the mechanism; and (3) arc image furnace experiments in which the plastics are exposed to temperatures approaching those of actual ablation applications.

Vapor pressure studies are being conducted using the Langmuir evaporation techniques. In addition matrix-isolation techniques and resonance-line absorption spectroscopy are being applied to the study of vapor species. Materials studied are tantalum carbide, zirconium carbide and thoria.

Normal spectral emissivities of tungsten, tantalum carbide and zirconium carbide have been measured between 1800°K and 3000°K using an emissometer designed and built for this purpose

Results of these studies are given and discussed.

PHYSICAL METALLURGY

WADC TR 59-655, OTS Release

April 1960

SUBJECT: RESEARCH AND DEVELOPMENT ON THE EFFECTS OF HIGH PRESSURE AND TEMPERATURE ON VARIOUS ELEMENTS AND BINARY ALLOYS
INVESTIGATOR: J. S. Harvey, L. Kaufman, S. A. Kulin, A. Leyenaar, H. Udin
CONTRACT: AF 33(616)-5996, Manufacturing Laboratories, Inc.
ABSTRACT: The accomplishments of the contract period July 1, 1958 to September 30, 1959 are summarized. High-pressure high-temperature apparatus capable of subjecting test specimens to 100,000 atmospheres pressure at temperatures in excess of 1500°C was designed and constructed. Investigations carried out in this apparatus demonstrate that high hydrostatic pressure acts to lower the temperature of martensitic transformations in iron-nickel alloys and expands the temperature range of the gamma loop in the iron-chromium system. The precipitation hardening process in both aluminum-copper and copper-beryllium alloys is significantly inhibited and modified when carried out at high hydrostatic pressure. The use of pressure during heat treatment of a series of carbon steels results in transformation products radically different in structure from those obtained by the same treatment at atmospheric pressure and also significantly retards the tempering reactions. Evaluation of mixtures of carbon in a ratio of 1:1 atomic percent with platinum, palladium and nickel which were subjected to pressures in excess of 80,000 atmospheres at approximately 3000°C reveals the existence of a new phase which is extremely hard and brittle and contains essentially no metal atoms. Preliminary experiments indicate that the Ti-N phase diagram is significantly modified by pressure.

WADC TR 59-674, OTS Release

April 1960

SUBJECT: THE PLASTIC COEFFICIENTS FOR WORK-HARDENING MATERIALS
INVESTIGATOR: W. H. Warner, P. Mahmoodi
CONTRACT: AF 33(616)-5449, University of Minnesota
ABSTRACT: In the establishment of the form of their generalized stress-strain law for work-hardening inelastic materials, Warner and Handelman did not show how the plastic coefficients could be evaluated in terms of material properties. This report first shows how this can be done, giving the general form of the coefficients for various states of stress. The validity of the assumptions behind this representation must be experimentally tested; comparison with existing results is carried out as far as possible. The conclusion suggests what types of experiments are needed to test the validity of the law, and if they are successful, what further experiments are needed to provide the information necessary to solve various combined stress problems analytically.

WADC TR 59-687, OTS Release

April 1960

SUBJECT: RESEARCHES ON HYDROGEN EVOLUTION
INVESTIGATOR: C. A. Knorr
CONTRACT: AF 61(052)-142, Technische Hochschule, Munich, Germany

WADC TR 59-687 (Continued)

ABSTRACT: Part I - Investigation of adsorption phenomena on platinum, rhodium, iridium, palladium, and gold, by means of the potentiostatic method of applying a triangular voltage. Formation and decomposition of hydrogen and oxygen cov-rages as a function of the potential. Determination of the retardation of charge transfers.

Part II - Investigation of the reactions of hydrogen occluded in Pd-wires with H-acceptors like chromic acid, potassium ferricyanide, and hydrogen peroxide, by the measurement of the longitudinal resistance. Study of the oxide and cyanide layers on the electrode surface with regard to the H₂ permeability and the influence of poisoning on the decomposition reaction.

WADC TR 59-747, OTS Release

July 1960

SUBJECT: RESEARCH AND DEVELOPMENT ON THE EFFECTS OF HIGH PRESSURE AND TEMPERATURE ON VARIOUS ELEMENTS AND BINARY ALLOYS

INVESTIGATOR: E. W. Goliber, K. H. McKee, J. S. Kasper, J. E. Hilliard, J. W. Cahn, V. A. Phillips

CONTRACT: AF 33(616)-5995, GE Metallurgical Products Department, General Electric Research Laboratory

ABSTRACT: An investigation has been made of the effect of pressure on the properties and kinetics of transformation in various alloy systems. Many of the experiments were exploratory and yielded no evidence for irreversible changes with pressures of up to 100,000 atm at elevated temperatures. However, a shift was observed in the gamma loop of the iron-chromium system from 12.5 to about 20 per cent chromium. Evidence for a similar shift in the gamma loop of the iron-aluminum system was also found. Pressure heat treatment of a tool steel gave a refinement of the austenite grain size and an improvement in the distribution of carbides.

An apparatus for X-ray diffraction analysis at pressures up to 35,000 atm has been constructed, and observations have been made on the bismuth I to II transition.

In the kinetic experiments it has been found that pressure markedly decreases the transformation rate of austenite to pearlite and the precipitation rate in the systems; aluminum-copper, copper-beryllium, lead-tin, and gold-nickel.

WADD TR 60-31, OTS Release

July 1960

SUBJECT: A STUDY OF THE EFFECTS OF SURFACE FILMS ON THE MECHANICAL PROPERTIES OF METALS

INVESTIGATOR: Irvin R. Kramer

CONTRACT: AF 33(616)-6220, The Martin Company

WADC TR 53-373, Sup 8

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WADD TR 60-31 (Continued)

ABSTRACT: It is shown that by removing the surface of aluminum single crystals while they are being deformed, the extent and slope of Stage I and Stage II are affected. The stress at which Stage III starts is also lowered. A dislocation pop-out phenomenon was observed when the rate of metal removed was suddenly increased during the tensile deformation. Deformed crystals of zinc, aluminum, and copper immersed in solutions containing surface active agents reacted with these agents to form metal soaps.

WADD TR 60-74, Part I, OTS Release

May 1960

SUBJECT: THE METALLURGY OF YTTRIUM AND THE RARE EARTH METALS
PART I. Phase Relationships
INVESTIGATOR: Bernard Love
CONTRACT: AF 33(616)-5905, Research Chemicals
ABSTRACT: Partial constitutional diagrams were established for sixteen binary systems containing rare earth metals. These studies were made as the first step in efforts to produce alloys with improved mechanical properties.

Erbium and yttrium form simple binary eutectic systems with titanium. No compounds are present. The transition temperature of titanium is not significantly affected. Copper, tin, and cobalt form intermetallic compounds with erbium and yttrium. The melting points of the rare earths are rapidly lowered, eutectics forming with the respective compounds. Vanadium forms extensive (and chromium forms limited) liquid immiscibility regions with erbium and yttrium. Eutectics are formed at the rare earth end of these systems. Terminal solubilities are low in all of the above systems.

The system erbium - zirconium is characterized by appreciable solubility of zirconium in erbium and extensive solubility of erbium in both alpha and beta zirconium. There are no inter-metallic compounds. The alpha erbium and beta zirconium solid solutions enter into a simple eutectic reaction. A high temperature peritectic reaction involving beta erbium is suggested. The system yttrium - zirconium is similar in all major respects except that the solubility limits at both ends of the system are somewhat lower. Preliminary investigation of the gadolinium - zirconium system also indicated similarity in all major respects.

Beryllium and ytterbium form an intermetallic compound. The terminal solubility of ytterbium in beryllium is low. No eutectic is observed at the beryllium end of the system.

The results obtained suggest zirconium as a promising alloying element. Evidence was also found for the purification of vanadium and beryllium when these metals were melted together with rare earths.

WADD TR 60-74, Part II

June 1960

SUBJECT: THE METALLURGY OF YTTRIUM AND THE RARE EARTH METALS
PART II. Mechanical Properties
INVESTIGATOR: Bernard Love
CONTRACT: AF 33(616)-5905, Research Chemicals
ABSTRACT: Study of the mechanical properties of rare earth metals was continued. Tensile, compression, fatigue, and impact properties were determined. Evaluation of the metals was extended to include hot and cold working characteristics, and studies were made of alloy systems predicted to have improved mechanical properties.

Cast yttrium, dysprosium, and erbium can be cold worked to improve mechanical properties. All are easily swaged at 980°C (1800°F). The resulting structures are uniform and dense, and the properties are improved over the cast condition. Tensile properties are quite comparable when the metals are in the same condition. Ductility (as measured by reduction of area) was somewhat limited for the cast structures, but was improved by working. The cast rare earth metals are notch sensitive. The fatigue properties follow the normal pattern of increased life with decreased maximum applied load. Extrapolated S-N curves indicate fatigue endurance limits in the order of 40-50% of the corresponding ultimate tensile strengths.

Zirconium enters into solid solution in erbium and results in marked tensile strengthening. Dispersed, second phase zirconium, also contributes to hardening.

The recrystallization behavior of erbium and yttrium follows a normal sequence of recovery, recrystallization and grain growth, complete recrystallization of erbium, cold worked 60%, occurs at approximately 1100°C; yttrium at approximately 900°C.

Studies were made of several methods for improving the purity of rare earth metals. Carbon deoxidation decreased the oxygen content slightly. Attempts to purify yttrium, erbium, and dysprosium by electron beam melting were not successful. Small quantities of both dysprosium and scandium were, however, successfully vacuum distilled at elevated temperatures. Analytic results indicate that the distilled metals are of significantly higher purity.

WADD TN 60-115, OTS Release

July 1960

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGES ON METALLIC SINGLE CRYSTALS: TIN
INVESTIGATOR: L. Peraldo Bicelli - A LaVecchia, Milano, Italy
CONTRACT: AF 61(052)-144
ABSTRACT: Hydrogen overvoltage on tin single crystal cathodes, oriented following the: (001), (100), (110) planes and on polycrystalline tin, has been measured in perchloric acid solutions with two different concentrations and in hydrochloric acid solutions.

Tafel law holds true; while the parameters are different for the different electrodes.

WADD TN 60-242, OTS Release

December 1960

SUBJECT: THE APPLICATION OF COMPUTER TECHNIQUES TO PREFERRED ORIENTATION STUDIES

INVESTIGATOR: J. R. Holland

ABSTRACT: A method has been devised and is currently being used to plot normalized pole figures by computer techniques. As the pole distribution traces, i.e., intensity of the diffracted X-ray beam, versus angular position of the specimen are not an acceptable input for the computer, the data required from such traces are entered onto punch cards. Corrections for defocusing effects may be readily made as the data are transferred from the Brown recorder traces to punch cards. It is possible to program other correction factors into the computer operations, providing that these correction factors or curves can be established accurately. The computer translates angular position of the specimen into rectilinear co-ordinates which are an identical representation of stereographic co-ordinates. This computer method has been used in conjunction with the Schulz reflection technique, but modification of the computer program permits its use with other quantitative X-ray techniques for determining preferred orientation.

The feasibility of using computer techniques to plot inverse pole figures has been considered.

WADD TN 60-269, OTS Release

February 1961

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: CADMIUM

INVESTIGATOR: L. Peraldo Bicelli, A. LaVecchia, N. Sala Cataffo

CONTRACT: AF 61(052)-144, Laboratori di Ellectrochimica, Milano, Italy

ABSTRACT: Hydrogen overvoltage on cadmium polycrystalline and single crystal cathodes, oriented following the (0001), (1010) and (1120) planes, has been measured in perchloric acid solutions.

The Tafel law holds nearly; the parameters being different for the different electrodes.

WADD TN 60-270, OTS Release

February 1961

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: NICKEL

INVESTIGATOR: L. Peraldo Bicelli, A. LaVecchia

CONTRACT: AF 61(052)-144, Laboratori di Ellectrochimica

ABSTRACT: Hydrogen overvoltage on nickel single crystal cathodes, oriented following the (100), (110) and (111) planes has been measured in perchloric acid, hydrochloric acid and in sulfamic acid solutions.

The Tafel law holds nearly; the parameters being different for the different electrodes.

WADD TR 60-275, OTS Release

October 1960

SUBJECT: HYDROGEN EMBRITTLEMENT OF TITANIUM ALLOYS
INVESTIGATOR: A. E. Riesen, D. H. Kah
ABSTRACT: The effect of various hydrogen interstitial contents between 60 parts per million and 312 ppm on the notch sensitivity and low strain rate embrittling of four titanium alloys was investigated at room temperature. The materials investigated included three alpha-beta alloys: Ti-6Al-4V, Ti-2Al-6Mo, and Ti-2.5Al-16V; and one all alpha alloy, Ti-5Al-2.5Sn, which were all heat treated per manufacturers recommended schedules. An optimum embrittling effect was encountered between 180 to 220 ppm hydrogen content in the alpha-beta alloys. Over the range of hydrogen content investigated the alpha alloy was not appreciably embrittled.

WADD TR 60-534, OTS Release

October 1960

SUBJECT: FIELD ION MICROSCOPY OF IRON WHISKERS
INVESTIGATOR: Erwin W. Muller
CONTRACT: AF 33(616)-6397, Pennsylvania State University
ABSTRACT: The design, construction and operation of a complete field ion microscope unit is described. The restrictions due to the high rate of field evaporation of iron are considered. This microscope is used for the study of the atomic structure of iron whiskers. Indications of crystal defects such as screw dislocations and interstitials are found, although the whiskers did usually not have sufficient strength to withstand the extremely high field forces.

WADD TR 60-543, OTS Release

October 1960

SUBJECT: ATTEMPTED SYNTHESIS OF BERYLLIUM HYDRIDE
INVESTIGATOR: John C. Powers, Donald W. Vose, Edward A. Sullivan
CONTRACT: AF 33(616)-5847, Research & Development Laboratories
ABSTRACT: The direct synthesis of beryllium hydrides from the elements was attempted in two sets of equipment designed to grind beryllium metal while subjected to heat and hydrogen pressure. Grinding action was supplied by mechanisms which provided the reaction vessels with either reciprocating vertical motion, or motion in which the reactor's horizontal axis described a circle. The ability of both mechanisms to grind metals satisfactorily was demonstrated in test runs, in which magnesium hydride was synthesized. However, beryllium hydride was not prepared by this method, even when catalytic additives were used. There were no indications even of partial hydriding.

Operation with beryllium, which is highly toxic, was carried out safely by conforming to recognized standards of industrial practice.

WADD TR 60-705, OTS Release

November 1960

SUBJECT: THE INFLUENCE OF PRESSURE AND TEMPERATURE ON ZIRCONIUM OXIDE

INVESTIGATOR: F. W. Vahldiek, C. T. Lynch, L. B. Robinson

ABSTRACT: The effects of temperature and pressure on the monoclinic-tetragonal phase transformation and on the sinterability of superpure zirconia have been studied. Investigations were undertaken at temperature ranges from 1140° to 1800°C and at pressures from 310 to 12380 atmospheres. Experiments showed that no tetragonal phase was stabilized after firing and rapid quenching in air, water and liquid nitrogen. The region of transformation was found between 1193° to 1200°C . Pressure lowered the sintering temperature of zirconia, and specimens pressed at 9280 atmospheres and fired to 1800°C in an inert atmosphere resulted in compacts with a 98.8% theoretical density.

Grain refinement was observed with increasing temperature and at various pressures. Average particle sizes of 4 microns and less were found in compacts originally consisting of particles averaging 60 microns. A mathematical equation for the compressibility of zirconia was derived.

WADD TR 60-769, OTS Release

February 1961

SUBJECT: RESEARCH ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS

INVESTIGATOR: R. Piontelli, L. Peraldo Bicelli, A. LaVecchia

CONTRACT: AF 61(052)-144, Politecnico di Milano, Milano, Italy

ABSTRACT: Hydrogen overvoltage on silver, lead, nickel, thin and cadmium single crystals has been measured in different experimental conditions. The results have been discussed and it has been observed that the Tafel law is generally followed.

MECHANICAL METALLURGY

WADC TR 56-585, Part II, OTS Release

September 1960

SUBJECT: EFFECTS OF TEMPERATURE-TIME-STRESS HISTORIES ON THE MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALLIC MATERIALS - PART II. Stressed Exposure of 7075-T6

INVESTIGATOR: C. D. Brownfield, D. M. Badger

CONTRACT: AF 33(616)-5769, Northrop Corporation

ABSTRACT: A study has been made on the problem of predicting strength of a hardened metal alloy after subjection to variable thermal and stress environments severe enough to cause permanent loss of properties. Methods have been developed for predicting tensile ultimate, tensile yield, and compressive yield strengths of 7075-T6 aluminum alloy after single or multiple exposure to various conditions of temperature and stress. An analytical expression suitable for automatic computing machine use has also been developed.

The results of tensile and compressive tests on alclad 7075-T6 aluminum alloy showed that stresses large enough to produce inelastic creep strain during thermal exposure cause reduction in residual strength after exposure. The test results have been used to establish the usefulness of the Larson-Miller exposure parameter for correlating residual strength after simple and complex, stressed and unstressed exposures.

WADC TR 57-649, Part II, OTS Release

April 1960

SUBJECT: DETERMINATION OF THE MECHANICAL PROPERTIES OF AIRCRAFT-STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING

INVESTIGATOR: James B. Preston, J. Robert Kattus

CONTRACT: AF 33(616)-3494, Southern Research Institute

ABSTRACT: Structural components in high-speed aircraft and in missiles must function for short periods of time at high temperatures and at high stresses, and frequently the heating and loading occur simultaneously. The requirements for reliability without overdesign demands accurate test data obtained under conditions approximating the expected operating conditions. In an effort to fulfill a portion of this need, this program was divided into four independent areas of work as follows: (1) The tensile properties of unalloyed beryllium were determined at test temperatures from ambient through 1500 F. (2) The short-time, elevated-temperature tensile properties were determined for ten combinations of base materials and coating materials (Cr-Ni electroplate on copper sheet, Rokide A on copper sheet, Rokide Z on copper sheet, Rokide ZS on copper sheet, Cr electroplate on A-nickel sheet, Ni-Cr electroplate on molybdenum sheet, Rokide Z on molded graphite, Rokide ZS on molded graphite, Crystalon C on molded graphite and SiC-SiN on molded graphite). (3) The effects of linear thermal gradients up to 1500 F/in. on the tensile properties of a typical refractory alloy were investigated. (4) The effects of simultaneous heating and loading on the tensile properties of a typical structural alloy were investigated. This report covers the first three of these four

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areas of work; a supplementary report will be issued shortly containing all of the results of the investigations involving simultaneously transient temperature and load.

WADC TR 59-416, Part II, OTS Release

July 1960

SUBJECT: INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES
PART II. Creep Buckling Experiments with Columns of
Ti-7Al-4Mo Titanium Alloy

INVESTIGATOR: Ralph Papirno, George Gerard

CONTRACT: AF 33(616)-5807, New York University

ABSTRACT: Creep buckling and short time buckling experiments have been performed on columns of Titanium Alloy Ti-7Al-4Mo with simulated simple support and fixed end boundary conditions. Both end shortening and central deflection were recorded autographically during loading and during creep. Elevated temperature properties of the test material had previously been determined from 3/16 inch thick stock and these were augmented by tests on 3/8 inch thick stock since the mechanical properties and structure were significantly different for the two thicknesses.

The excellent creep properties of the test material indicate that creep buckling tests must be conducted at 950°F or higher for creep buckling to occur in reasonable times at stress levels below the short time compressive yield strength. Since only a narrow range of test conditions is available for creep buckling experiments with this material, column tests were conducted at L'P of approximately 40 at 950°F.

WADC TR 59-511, OTS Release

May 1960

SUBJECT: GAS ATMOSPHERE EFFECTS ON MATERIALS

INVESTIGATOR: R. A. Baughman

CONTRACT: AF 33(616)-5667, General Electric Company

ABSTRACT: A three phase program was conducted to determine the effect of a special gas containing 5% H₂-95% N₂ on the properties of several materials. Both low and high temperature materials were included. The gas was about 70% saturated with water vapor. The following items were included:

1. Elevated temperature investigation including 6 high temperature alloys for tensile tests, rupture tests and chemical analysis. Two systems of gas atmosphere testing was included on A-286, L-605, X-40, R-41, Inco 702 and U-500. Limited tests were made on welded, brazed and notched specimens.

2. Low temperature investigation including seven low temperature alloys for tensile tests, strain aging tests and impact tests. The alloys included were 302 SS, 316 SS, 6061, Al10AT, Be-Cu, R-41 and 17-7PH. Limited tests were included on welded and brazed specimens.

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3. Fundamental mechanism and coating study was done on Inco 702, which is most sensitive to gas environment. Special oxidation cycles, over-aging cycles, strain aging cycles, specimen geometric changes and coatings were tested by rupture techniques to indicate the nature of the mechanism reducing rupture strength and possibilities for preventing strength loss.

This report includes all data obtained and detailed discussion of results. The high temperature rupture strength of each alloy studied was shown to be reduced below the corresponding air atmosphere strength by the special gas. The low temperature properties were shown to be independent of the environmental effects studied. The basic mechanism effecting the high temperature properties has not been clearly determined.

WADC TR 59-762, Part II, OTS Release

May 1960

SUBJECT: ULTRA-SHORT-TIME CREEP RUPTURE
INVESTIGATOR: Joseph S. Ives, Jr.
CONTRACT: AF 33(616)-5557, American Machine & Foundry Company
ABSTRACT: The creep behavior of eight structural sheet metals, (321 stainless, 410 stainless, Inconel-X, PH-15-7 Mo, Rene 41, Udimet-500, B120 VCA Ti, and AM 350), when subjected to temperatures up to 2,000°F within 200 milliseconds is presented in tabular and graphic form. The equipment used for these measurements consisted of a Tatnall Model RL-6 Creep machine modified so that a 2000 microfarad bank of capacitors, charged to 5000 volts could be discharged through the test specimen to bring it to the desired test temperature. This initial discharge was followed by an ac current of sufficient magnitude to maintain the specimen at the test temperature.

Data was taken on each of the materials at four creep rates for each temperature. From this data strain vs time, isochronous stress vs strain, and stress vs time curves were plotted and are presented in the Appendix. Data was not reported when an improper transfer of energy from the capacitors to the specimens occurred. Emissivity data, or data from which the emissivity can be calculated, for each of the materials are presented. In addition, the initial transient behavior, due to thermal expansion, of the specimens is given in tabular form.

WADC TR 59-762, Part III, OTS Release

August 1960

SUBJECT: ULTRA-SHORT-TIME CREEP RUPTURE
INVESTIGATOR: C. W. H. Barnett
CONTRACT: AF 33(616)-6798, American Machine & Foundry Company
ABSTRACT: The creep behavior of three structural sheet materials, namely HM-21, H-11 and 310 stainless steel, was studied to temperatures up to 2,000°F within 200 milliseconds. The recorded results are reported in tabular and graphic

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form. The testing procedure used in implementation of previous contracts for this same type of work has been modified to impose the stress on the material simultaneously with the heating action. In the previous work, on the other hand, the stress was first applied following which step the specimen was heated.

The equipment used for all tests consists of a Tatnall Model RL-6 creep machine, so modified that a charge in a bank of capacitors can be used to heat the test specimen to the desired temperature by controlling the voltage of the capacitor charge.

Data were taken on each of the materials at four creep rates and at three loading rates. The controlled loading rates are an innovation in this contract. From the data obtained, curves of strain vs time, isochronous stress vs strain, and stress vs time were plotted and are presented in the Appendix.

WADD TR 60-42, OTS Release

July 1960

SUBJECT: SOME QUANTITATIVE ASPECTS OF FATIGUE OF MATERIALS
INVESTIGATOR: Harold N. Cummings
CONTRACT: AF 33(616)-6552, Curtiss-Wright Corporation
ABSTRACT: In this report are given not only the fatigue properties of many structural materials but also the "static" properties and such other supplementary information as was given in the references consulted. The data are in general from room temperature tests, but a few data are given on tests at higher temperatures. The data are presented in tables and on curves, supplemented by brief discussions in the text.

WADD TR 60-53, OTS Release

June 1960

SUBJECT: EFFECT OF TEMPERATURE ON THE CREEP OF POLYCRYSTALLINE ALUMINUM BY THE CROSS-SLIP MECHANISM
INVESTIGATOR: N. Jaffe, J. E. Dorn
CONTRACT: AF 33(616)-3860, University of California
ABSTRACT: The apparent activation energy for creep of polycrystalline aluminum was determined over the range of 273° to 350°K by the effect of small abrupt changes in temperature on the creep rate. A constant activation energy of $27,400 \pm 1000$ cal/mole was obtained over strains of 0.003 to 0.23, stresses ranging from 2250 to 6000 p.s.i. and strain rates varying from 0.1145×10^{-5} to 29.5×10^{-5} per minute. Metallographic studies and comparison with theory suggested that creep in this range is controlled by the rate of cross-slip of dislocations.

Both X-ray diffraction analyses and room temperature tensile stress-strain data following precreep revealed that the substructure produced in

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this range by creep under a given stress depended only on the total creep strain being independent of the actual test temperature. As a result it was deduced that the total strain ϵ during creep under a given stress should be a function of temperature compensated time $\Theta = te^{-Q/RT}$, energy, R the gas constant and T the absolute temperature. A number of creep tests conducted at two different temperatures verified the validity of this conclusion.

WADD TR 60-60, OTS Release

May 1960

SUBJECT: STEADY STATE RESPONSE OF BEAMS WITH TRANSLATIONAL AND ROTATIONAL DAMPING MOTIONS AT THE SUPPORTS
INVESTIGATOR: C. C. Fu, T. J. Mentel
CONTRACT: AF 33(616)-5426, University of Minnesota
ABSTRACT: Two methods of analysis are presented for the steady state response of beams with translational and rotational damping motions at the supports. The first of these methods uses a continuous model and the second uses a discrete (three-degree-of-freedom) model. Both cases are characterized by non-linear equations and approximate solutions are produced for each. Numerical results are presented, giving comparisons and optimum damping configurations.

WADD TR 60-161, Part I, OTS Release

November 1960

SUBJECT: EXPERIMENTS ON SLIP DAMPING AT ROUNDED CONTACTS
INVESTIGATOR: L. E. Goodman and G. E. Bowie
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: This report describes an apparatus for studying slip damping at the contacts of a sphere with two flat parallel plates. A load of constant magnitude is applied normal to the plates and the sphere is driven in a direction tangential to the plates by an electrostrictive device operated at 60 cycles per second. Experimental results obtained with a one half inch diameter type 316 stainless steel sphere pressed between flats of the same material are presented. They are in fair agreement with theoretical predictions by R. D. Mindlin, et al. Results for pyrex glass differ from those for stainless steel and are not compared with theory. Suggestions are made for future work on contact problems.

WADD TR 60-188, OTS Release

July 1960

SUBJECT: INFLUENCE OF NATURAL FREQUENCIES AND SOURCE CORRELATION FIELDS ON RANDOM RESPONSE OF PANELS
INVESTIGATOR: R. F. Lambert, D. H. Tack
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: Theoretical and experimental analysis of random responses of panels with a view toward problems of design are presented. The influence

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of source correlation fields, damping, and boundary impedance are integrated into the analysis. Modal response is viewed from the complex frequency plane in the form of a pole diagram. The concepts developed from this point of view have direct application in design for specified mean square response. The statistical independence of modes and its relationship to properties of the source and panel are discussed and interpreted for several situations.

WADD TR 60-191, OTS Release

May 1960

SUBJECT: THE DETERMINATION OF THE EFFECTS OF ELEVATED TEMPERATURES
ON THE STRESS CORROSION BEHAVIOR OF STRUCTURAL MATERIALS

INVESTIGATOR: Frank A. Crossley, Carl J. Reichel, Charles R. Simcoe

CONTRACT: AF 33(616)-6392, Armour Research Foundation

ABSTRACT: Certain aspects of elevated-temperature chloride salt stress-corrosion of Ti-6Al-4V alloy have been investigated. Experiments were conducted to determine whether the protection of the oxide film is lost by direct chemical reaction, or by dissolution, i.e., oxygen diffusion into the base metal. The dissolution rates of anodized oxide films over the temperature range from 800° to 1000°F were determined. Coefficients for oxygen diffusion in Ti-6Al-4V alloy are given by:

$$D, \text{ cm}^2/\text{sec} = 2.87 \times 10^4 \exp (-67,900 \pm 300/RT).$$

Experiments showed conclusively that film protection is lost by chemical reaction of the chloride salt with TiO_2 .

Elevated-temperature creep testing in the presence of chloride salt was found to be detrimental to post-exposure tensile properties of the following materials: 17-7 PH, stainless steel Type 321, Inconel-X, and B120-VCA titanium alloy. Materials which, apparently, were not harmed by such exposure were: 2024T-86 aluminum alloy, Nickel-plated 17-22 AS low alloy steel, Lapelloy 13% chromium steel, and ZK-60 magnesium alloy.

Materials exhibiting premature failure with greatly reduced ductility in tension static fatigue tests at room temperature in the presence of 10% chloride salt aqueous solution were: 2024T-86, ZK-60, 17-7 PH, and B120-VCA.

The presence of JP-4 fuel in tension static fatigue tests at room temperature was not indicated to be detrimental to 2024T-86, Nickel-plated 17-22 AS, 17-7 PH, Lapelloy, Type 321, or B120-VCA. There was some indication that the presence of fuel residue in elevated-temperature creep tests may be slightly detrimental to 2024T-86 alloy.

SUBJECT: ELEVATED TEMPERATURE DYNAMIC MODULI OF VANADIUM TITANIUM AND V-TI ALLOYS

INVESTIGATOR: W. H. Hill, B. A. Wilcox

ABSTRACT: The dynamic moduli of calcium-reduced vanadium, high grade aluminothermic vanadium, iodide titanium and Ti-75A alloy were determined over the temperature range R. T. to 1250°F. In addition, elevated temperature dynamic modulus data were obtained for five V-Ti alloys, having nominal compositions of V-8Ti, V-17Ti, V-25Ti, V-32Ti, and V-48Ti. The decrease in modulus of vanadium with increasing temperature was found to be relatively small as compared to iodide Ti and Ti-75A. Solid solution additions of Ti were observed to lower the modulus of V over the entire temperature range studied, but the additions did not affect the slope of the modulus versus temperature curve.

An empirical correlation relating modulus to density, absolute melting point, and atomic weight was successfully applied to vanadium and V-Ti alloys, such that good agreement was realized between the ratios of E_v/E alloy (calculated) and E_v/E alloy (measured).

SUBJECT: THE EVALUATION OF THE EFFECTS OF VERY LOW TEMPERATURES ON THE PROPERTIES OF AIRCRAFT AND MISSILE METALS

INVESTIGATOR: Leonard P. Rice, James E. Campbell, Ward F. Simmons

CONTRACT: AF 33(616)-6345, Battelle Memorial Institute

ABSTRACT: In the past few years, the use of liquefied gases such as oxygen, nitrogen, hydrogen, and helium has increased enormously in the aircraft and missile industries. There is a great deal of interest in metallic materials to store, transport, or otherwise handle these extremely low-temperature liquids. This investigation presents the tensile and hardness properties of eight different alloys of interest to the aircraft and missile industries at temperatures ranging from -253°C (liquid hydrogen) to room temperature. These alloys are Ti-6Al-4V, Ti-4Al-3Mo-IV, Ti-16V-2.5Al, and B-120VCA (all-beta alloy) titanium alloys; 17-7PH, PH15-7Mo, and Type 301XH stainless steels; and Vascojet 1000 alloy steel.

In general, the values for hardness, elastic modulus, and tensile and yield strengths of these materials tended to increase as the best temperature was reduced to -253°C. However, of the eight alloys investigated, the Ti-6Al-4V and the Ti-4Al-3Mo-IV titanium alloys and the Type 301XH stainless steel were the only ones that did not reveal a serious loss of ductility or did not fracture before reaching 0.2 per cent offset strain at -253°C. In most of the other alloys, brittle behavior, as revealed by notched tensile tests, became evident at temperatures of -78°C and below. The titanium alloys have the highest yield strength-density ratios at low temperatures of any of the materials tested.

WADD TR 60-278, OTS Release

September 1960

SUBJECT: NOTCH SENSITIVITY OF REFRACTORY METALS
INVESTIGATOR: Albert G. Ingram, Frank C. Holden, Horace R. Ogden,
Robert I. Jaffee
CONTRACT: AF 33(616)-6291, Battelle Memorial Institute
ABSTRACT: The tensile and notch tensile properties of four refractory metals, molybdenum, tungsten, columbium, and tantalum, and one alloy, Mo-0.5 Ti, were investigated at five temperatures selected to encompass the brittle-to-ductile transition. All specimen failures were classified according to fracture mechanism. The notch sensitivity of each material was evaluated by analyzing the notch and unnotch tensile strength, the ductility transition, and the fracture transition.

WADD TR 60-280, OTS Release

June 1960

SUBJECT: RHEOLOGICAL PROPERTIES OF ADHESIVES CONSIDERED FOR INTERFACE DAMPING
INVESTIGATOR: J. S. Whittier
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: Machines for testing soft adhesives under static compression and shear and under dynamic shear are described. Static creep data in compression and shear are reported for Minnesota Mining and Manufacturing Company's 3M Tape #466. At a given compressive load this material deforms, after sufficient time has elapsed to an "equilibrium" thickness. Dynamic shear data for 3M Tape #466 are reported for frequencies from 0.1 to 120 cps and for shear strain amplitudes from zero to unity and greater. Stress history and fatigue effects are also mentioned. This material is found to dissipate large amounts of specific damping energy when undergoing safe dynamic shear strains. Spot checks on the dynamic shear properties of three other materials at 11.5 cps are also presented. The terms used for data presentation are explained in the report by giving data reduction formulas.

WADD TR 60-308, OTS Release

August 1960

SUBJECT: QUASI-ORTHOGONAL MODES OF DYNAMICAL SYSTEMS
INVESTIGATOR: Lawrence E. Goodman, Yellappa C. Das
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: When energy dissipating devices are inserted at the boundaries the analysis of structural vibration problems becomes difficult. The usual classical methods in such cases yield an infinite set of equations for the coefficients of a modal expansion of the solution. The method developed in this report, gives a finite equation for each coefficient of the modal expansion and thus makes it possible to obtain solutions for structural vibration problems with boundary conditions involving the time derivatives.

The structural vibration problem treated is identical with that of the transient analysis of electrical circuits having distributed capacity and inductance and terminated by a lumped resistance. Certain problems in heat conduction also fall within the scope of the method developed.

WADD TR 60-309, OTS Release

August 1960

SUBJECT: SUPERSONIC SPEED FLUTTER ANALYSIS OF CIRCULAR PANELS WITH EDGES ELASTICALLY RESTRAINED AGAINST ROTATION
INVESTIGATOR: L. E. Goodman, Jasti Venkata Rattayya
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: The flutter problem is formulated, in terms of small-deflection plate theory, for a flat circular panel with edges elastically restrained against rotation. The panel is taken to be subjected to isotropic tension or compression in its middle plane. Linear piston theory is used to predict the aerodynamic load on the vibrating panel due to a supersonic compressible flow passing its upper surface.

Results of the analysis predict panel thickness necessary to prevent flutter as a function of air speed, elevation, and degree of edge fixity. Investigations of aerodynamic and structural damping effects and the influence of mid-plane load are included.

WADD TR 60-310, OTS Release

September 1960

SUBJECT: THE EFFECT OF SEVERAL GEOMETRICAL VARIABLES ON THE NOTCH TENSILE STRENGTH OF 4340 STEEL SHEET HEAT TREATED TO THREE STRENGTH LEVELS
INVESTIGATOR: Volker Weiss, John Sessler, Paul Packman, George Sachs
CONTRACT: AF 33(616)-6523, Syracuse University Research Institute
ABSTRACT: A systematic study was conducted to evaluate the effects of initial stress distribution and strength level on the notch tensile behavior of a typical high strength steel sheet alloy (SAE 4340) in the presence of stress concentrations.

The initial stress distribution, characterized by the stress concentration factor (K) and the stress gradient at the root of a notch, was determined analytically from the notch geometry according to elastic theory. As part of the study, K was kept constant and the stress gradient allowed vary for different notch depths and section widths.

The 4340 steel alloy sheet was tested in three different heat treat conditions ranging from ductile ($F_{tu} = 150$ ksi) to moderately notch brittle ($F_{tu} = 260$ ksi).

For the material conditions investigated, it was found that the effect of stress gradient on notch strength was fundamentally identical with that of section width. Increasing the section width (or decreasing the stress gradient) resulted in a decrease in notch strength when all other factors were held constant. However, the effect was small when compared to the section size effect observed previously for notched cylindrical bars.

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In addition, results of tests on an extremely brittle titanium sheet alloy indicated that the stress gradient is the predominant factor that influences notch strength. For a truly brittle material (which exhibits no plastic deformation prior to fracture), it appears that the stress concentration factor, the depth of a notch and the specimen width are significant only insofar as they contribute to the magnitude of the stress gradient and the maximum stress at the notch root

WADD TR 60-313, OTS Release

April 1960

SUBJECT: RESEARCH ON THE MECHANISMS OF FATIGUE
INVESTIGATOR: J. C. Grosskreutz
CONTRACT: AF 33(616)-6383, Midwest Research Institute
ABSTRACT: Based on a critical appraisal of existing fatigue theory and experimental data, significant research areas have been outlined for the study of fatigue mechanisms. Polycrystalline aluminum and copper and single crystals of aluminum have been used to investigate dislocation densities and configuration during crack initiation and the mechanism by which cracks propagate, both on the surface of a sample and internally. Back reflection X-ray patterns show that an observable increase in dislocation density occurs within the first 0.1 percent of fatigue life and that a saturation density is reached after only 1 percent of the life. These dislocations accumulate into stable subgrain boundaries provided either that the stressing is done under a tensile preload or that the amplitude of symmetrical stressing is large enough. For small symmetrical loading (strains ~ 0.001 for Al) a random dislocation array results with no subgrain formation. These results can be correlated with fatigue hardening behavior and possibly with the shape of the S-N curve. There is no obvious relation between the bulk behavior of fatigue induced dislocations and the formation of the initial fatigue crack which is a highly localized phenomenon.

Continuous observation of the surface of fatiguing samples utilizing strobe-microphotography has confirmed the accumulating evidence that cracks initiate in persistent slip bands. Tapered sections have shown that these slip bands correspond to deep notches or fissures. The propagation of the crack is observed to occur along well defined slip systems with the crack jumping from one parallel slip band to another by cross slip. Transverse sections cut through the crack have shown that the crack propagates into the volume of the metal from the surface, again via slip planes. The crack is observed to propagate generally perpendicular to the stress axis; with the available slip system, this usually results in a zigzag path. A "cloud" of slip is observed to precede the crack tip and from this fact and the foregoing data a mechanism of crack propagation is postulated; i.e., crack propagation is a series of re-initiations in persistent slip bands.

WADD TR 60-326, OTS Release

May 1960

SUBJECT: THE EFFECT OF DECREASES IN STRESS ON THE CREEP BEHAVIOR OF POLYCRYSTALLINE ALUMINUM IN THE DISLOCATION CLIMB REGION

INVESTIGATOR: L. Raymond, W. Ludemann, N. Jaffe, J. E. Dorn

CONTRACT: AF 33(616)-3860, University of California

ABSTRACT: The effect of stress on the creep rate in the dislocation climb region was isolated from the effect of substructural changes by determining the effect of decreasing the stress at fixed creep strains on the subsequent course of creep. Two substructures, one produced by precreeping at 1300 psi to a strain early in the primary stage and another produced by precreeping to the secondary stage at the same stress, were studied. It was found that the effect of stress on the creep rate was a function of the substructure; the creep rate increases less rapidly with stress for the substructure present at the secondary creep stage which contains more closely spaced barriers to dislocation motion. Whereas the barriers to slip are introduced during the primary stage of creep in the dislocation climb region, these barriers are thermally recoverable. The secondary stage of creep is reached when the rate of introduction of new barriers equals the rate of removal of existing barriers as a result of recovery. Due to recovery of barriers the same secondary creep rate is eventually obtained for a given final stress regardless of the preceding creep history.

WADD TR 60-360, OTS Release

July 1960

SUBJECT: EFFECT OF VISCOELASTIC FOUNDATION ON FORCED VIBRATION OF LOADED RECTANGULAR PLATES

INVESTIGATOR: C. T. Hsu, C. W. Chu, C. C. Chang

CONTRACT: AF 33(616)-6828, University of Minnesota

ABSTRACT: Forced vibration of a loaded rectangular plate with visco-elastic foundation is treated. Two types of visco-elastic material are considered, Kelvin-Voigt solid and Boltzmann model. Analytic solutions are given for the plate subject to compression and a periodic transverse pressure. A numerical example is worked out for the case of Kelvin-Voigt solid. Some features revealed by the examples are:

a. With visco-elastic effect, the natural vibration damps out rapidly (Fig. 3-5).

b. There exists a critical damping factor ψ_c . For $\psi < \psi_c$ the damping is oscillatory (Fig. 3); otherwise the damping is monotonic (Figs. 4-5).

c. The closer ψ is to ψ_c in magnitude, the faster the damping.

d. The time required for damping is almost independent of the frequency of the periodic loading.

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e. The greater the viscosity coefficient μ_f , the smaller the amplitude of vibration.

The above statement is obtained from preliminary computations. Further work has to be done before conclusive summary can be made.

WADD TR 60-403, OTS Release

December 1960

SUBJECT: DEVELOPMENT OF RANDOMLY ORIENTED WROUGHT BERYLLIUM SHEET
INVESTIGATOR: F. M. Yans, A. K. Wolff, A. R. Kaufmann
CONTRACT: AF 33(616)-6616, Nuclear Metals, Inc
ABSTRACT: Various factors affecting texture development in beryllium were studied in an effort to produce randomly oriented wrought beryllium sheet. An extensive literature survey was performed, the purpose of which was to ascertain the major phenomena affecting textural changes in metals, especially beryllium. Texture analysis techniques applicable to the determination of textures in beryllium sheet were evaluated and subsequently standardized. A combination of the original and modified Schulz techniques was chosen as the best method of evaluating the texture of a beryllium sheet sample.

Rolling experiments indicated that the specimen geometry and rolling sequence affected the textures developed during working. It was determined that, during rolling, the basal plane population parallel to the plane of the sheet increases with reduction in area and, when certain specimens are heat treated at high temperatures for short times, the basal plane population parallel to the plane of the sheet is reduced. Further rolling and annealing studies performed on bi-directionally rolled sheet indicated that, when the sheet is subjected to annealing temperatures in excess of 950°C , the location of the basal plane peak intensity is shifted and the intensity profile changes considerably, yielding a third-dimensional ductility of 1.75%. It should be noted that the same sheet in only the stress-relieved condition possesses only 0.2% third-dimensional ductility. The same textural changes were observed in sheet samples manufactured by compression rolling.

BeO particles were dispersed in a matrix of beryllium prior to working and the turbulent flow caused by the presence of these BeO particles reduced the degree of preferred orientation developed for a given working procedure. However, the embrittling effect of the BeO dispersion offset any improvements in properties resulting from the decrease in preferred orientation.

Single crystal rolling and annealing studies, in combination with hot-stage microscopy, further corroborated the fact that twinning is a

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primary mode of deformation in beryllium sheet. Twin absorption, recrystallization, and grain formation were also studied in the single crystals. It was noted that recrystallization in beryllium can occur at temperatures as low as 1200°F.

WADD TR 60-426, OTS Release

July 1960

SUBJECT: FATIGUE AND STRESS RUPTURE PROPERTIES OF INCONEL 713C, V-57C AND TITANIUM ALLOYS 7Al-3Mo-Ti AND MST 821 (8Al-2Cb-1Ta-Ti)
INVESTIGATOR: A. E. Cers, A. A. Blatherwick
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: Fatigue, rupture and creep data at various temperatures obtained at various alternating and mean stress combinations are presented for the alloys Inconel 713C, V-57C (modified Super A-286), 7Al-3Mo-Ti and MST 821 (8Al-2Cb-1Ta-Ti). The tests were performed on unnotched specimens and for Inconel 713C also on notched specimens having a theoretical stress concentration factor of 2.9. The data are presented as S-N curves and stress range diagrams to show the effect of temperature, ratio of alternating-to-mean stress, stress magnitude and specimen notch on the fatigue and rupture properties.

WADD TR 60-437, OTS Release

December 1960

SUBJECT: EFFECT OF STRESS NONLINEARITY ON EXTREMAL STATISTICS AND FATIGUE LIFE OF A SIMPLY SUPPORTED BAR
INVESTIGATOR: Richard H. Lyon
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: The statistics of the extrema of the stress at the surface of a supported bar are studied when the lowest mode of the bar is excited by random noise. The stress is a nonlinear function of displacement because the hinged ends have a fixed separation. The effect of this nonlinearity on the distribution of maxima and minima is calculated assuming that the tensile stress does not significantly affect the displacement statistics. The effect of nonlinearity on expected fatigue life is calculated for a linear fatigue law. It is found that nonlinearity may significantly diminish the expected fatigue life.

WADD TR 60-451, OTS Release

November 1960

SUBJECT: EFFECT OF THERMAL-MECHANICAL VARIABLES ON THE PROPERTIES OF MOLYBDENUM ALLOYS
INVESTIGATOR: M. Semchyshen, Robert Q. Barr, Gordon D. McArdle

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WADD TR 60-451 (Continued)

CONTRACT: AF 33(616)-5447, Climax Molybdenum Company of Michigan
ABSTRACT: Unalloyed molybdenum and three alloys, Mo-0.50% Ti, Mo-0.05% Zr, and Mo-0.49% Ti-0.057% Zr, have been studied from the standpoint of effect of fabrication variables on mechanical properties and structural stability. Strain hardening was accomplished either by rolling or by forging. Working temperatures were 2200 and 3000°F.

Molybdenum and the above-mentioned alloys were capable of being strain hardened even at low reductions by rolling or forging at 3000°F. Thus none of the reductions studied fell under the scope of true "hot-working". A previously observed contradiction to classic concepts was again observed in that bars reduced up to about 30% in area at 3000°F exhibited higher hardnesses and higher tensile strengths than bars reduced similar amounts at 2200°F. At higher reductions, recrystallization during the reduction schedule entered the picture, with the result that bars rolled at 2200°F displayed the higher hardness and strength values.

For any given set of deformation variables, the highest strength properties and greatest resistance to recrystallization were consistently demonstrated by the ternary alloy, Mo-0.49% Ti-0.057% Zr.

The role of externally applied stresses in influencing the recrystallization behavior of the materials under investigation was found to be quite complex. At least part of the complexity of the problem as approached here resulted from the large number of fabrication variables that were inherent in the test specimens. Depending upon the magnitude of the externally applied stress, it was possible either to lower or to raise the hardness as compared with similarly exposed, unstressed specimens.

Four new experimental alloy compositions are discussed. The extrusion of these complex molybdenum-base alloy castings was greatly facilitated by the use of ceramic (Al_2O_3) coated extrusion dies.

WADD TR 60-468, OTS Release

August 1960

SUBJECT: HIGH VELOCITY ELECTRIC ACCELERATOR SYSTEMS
INVESTIGATOR: Jordan J. Baruch, Denis U. Noiseux, Jay H. Ball, C. M. Gogos
CONTRACT: AF 33(616)-5730, Bolt Beranek and Newman, Inc
ABSTRACT: Accelerators designed to convert electrical energy to particulate kinetic energy have been investigated and their capabilities and limitations ascertained. Experimentally, speeds up to 14,500 ft/sec have been obtained with a 20,000 joule energy input. Four different mechanisms for electrical-kinetic energy conversion have been worked out and tested experimentally.

WADD TR 60-523, OTS Release

October 1960

SUBJECT: APPLICABILITY OF PRESENT CREEP PREDICTION TECHNIQUES FOR EXTRAPOLATING VERY LONG TIME CREEP BEHAVIOR

INVESTIGATOR: K. D. Shimmin

ABSTRACT: A number of methods of correlation and extrapolation of creep and stress-rupture data are reviewed and discussed briefly. It is shown that, of the time-temperature parameter correlation methods, the Manson-Haferd parameter method has been most successful in correlating existing data. Of the empirical methods of extrapolation, the graphical method of Grant and Bucklin, and the statistical approach reported by Clauss are shown to hold promise as a means of predicting long-time creep data. Recommendations are given for an experiment to provide verification and evaluation of the methods of extrapolation of long-time data.

WADD TR 60-553, OTS Release

October 1960

SUBJECT: APPLICATION OF ULTRASONICS TO SOLID ROCKET SYSTEMS

INVESTIGATOR: R. E. Kleint, R. D. McKown, J. B. Ramsey

CONTRACT: AF 33(616)-6603, Ultrasonic Testing & Research Laboratory

ABSTRACT: This report contains the results of investigations to establish the applicability of ultrasonic inspection techniques to solid fuel rocket motors. Special emphasis was placed on the detection of unbonded areas at the interfaces of solid fuel rocket motors having a basic construction of a steel case, rubber liner and solid propellant bonded together in that order. The acoustic properties were obtained for these and other materials used for construction of rocket motors. Commercially available ultrasonic inspection and recording equipment was used to obtain data. Various pulse-echo and through-transmission techniques were utilized to determine optimum conditions for detecting defects and displaying them on both cathode ray image and C-Scan (plan view) facsimile paper recordings.

WADD TR 60-580, Part II, OTS Release

January 1961

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS - Part II. The Effect of End Conditions on the Collapse Load of Columns

INVESTIGATOR: G. A. Costello, O. M. Sidebottom, Eugene Pocs

CONTRACT: AF 33(616)-5658, University of Illinois

ABSTRACT: A theory was presented for constructing the load-deflection relation and for determining the collapse load of a column having any known end condition. A trial and error solution was required which used interaction curves and assumed that the inelastic column assumed the shape of a sine curve. For time independent (creep) inelastic deformation arc hyperbolic sine interaction curves were used.

The experimental part of the investigation included tests of rectangular section columns made of 17-7PH stainless steel and tested at

WADD TR 60-580, Part II (Continued)

room temperature and at 972°F. Several slenderness ratios were considered, and the columns had end conditions which were either fixed, equal and opposite end eccentricities, or unequal end eccentricities. Good agreement was found between theory and experiment.

WADD TR 60-580, Part III, OTS Release

January 1961

SUBJECT: INELASTIC DESIGN OF LOAD CARRYING MEMBERS - Part III. The Significance of an Inelastic Analysis of Eccentrically-Loaded Members
INVESTIGATOR: O. M. Sidebottom
CONTRACT: AF 33(616)-5658, The University of Illinois
ABSTRACT: The author has worked with others on ten investigations, sponsored by Wright Air Development Division, which have considered the theoretical and experimental inelastic analyses of eccentrically-loaded tension and compression members. In all cases good agreement was found between theory and experiment for members tested at room temperature and at elevated temperatures. This investigation was undertaken to consider the significance of an inelastic analysis of eccentrically-loaded members. If the inelastic deformation can be considered time independent, a choice has to be made between an elastic and an inelastic solution. A study was made of the effect of several variables on the ratio of the load necessary to produce a specified inelastic deformation to the maximum elastic load. If the inelastic deformation is time dependent (creep), the only choice is an inelastic solution.

WADD TR 60-752, OTS Release

February 1961

SUBJECT: REDUCTION OF THE ENDURANCE LIMIT AS A RESULT OF STRESS INTERACTION IN FATIGUE.
INVESTIGATOR: Robert A. Heller
CONTRACT: AF 33(616)-7042, Columbia University
ABSTRACT: This paper presents the results of an investigation of the effects of stress interaction on fatigue life of aircraft structural materials subjected to randomized load spectra. All three materials: 2024 and 7075 aluminum and SAE 4340 steel exhibit fatigue lives shorter than those predicted on the basis of the linear (Miner) damage rule. A quasi-linear rule is proposed with a variable, spectrum dependent, endurance limit producing safe life estimates; the dependence of the endurance limit on the stress spectrum and its resulting design inadequacy is shown.

Tests were performed on high speed, programmed rotating bending fatigue machines of special design.

SUBJECT: EXPERIMENTATION, ANALYSIS AND PREDICTION FOR ENVIRONMENTAL CREEP
INVESTIGATOR: C. J. Giemza
CONTRACT: AF 33(616)-6453, The Martin Company
ABSTRACT: An examination was made of the environmental creep behavior of bare 7075-T6 aluminum alloy sheet with the aim of developing an experimental approach as well as relations for characterizing a material's response to an arbitrary stress-temperature-time environment following a prior history during which a metallurgical change has occurred.

A technique called Random Balance (Ref. 2), partly modified to a multiple balance design, was employed to select from several million possible environment test combinations a representative group. The general employment of associated techniques for analysis, common to the statistical field, was precluded because of the creep scatter which was attributed to inhomogeneity in the aluminum sheet.

It was found that a loss of strength in the order of 5% caused by overaging could result in an increase of total creep strain of several hundred percent in subsequent cycles. However, it was also observed that a recovery mechanism, following overaging, could restore a significant degree of resistance to creep.

In every instance, it was found that a transient stage of creep was manifested in each cycle upon reapplication of load. The magnitude of transient creep strain, as well as duration of the transient stage after the initial cycle, diminished as the number of cycles increased for combinations of high stress and high temperature or low stress and low temperature. At test conditions which could be identified with a service environment, the transient creep stage comprised a relatively large fraction of the total creep strain generated during a given cycle.

A qualitative association of creep behavior of the specimens to their origin in the aluminum sheet revealed a pronounced inhomogeneity which could not be related to static tensile properties. Furthermore, small creep strain differences which specimens exhibited at moderate stresses were magnified during subsequent recycling as the magnitudes of stress and temperature increased; slight overaging further severely aggravated the initial disparities among the specimens.

Excellent reproducibility of an arbitrary test environment was possible when the repeat tests were made with specimens which were obtained from adjoining positions in the sheet. Accordingly, as the distances between specimens in the sheet increased, the differences in creep behavior became greater for identical tests, regardless of nominal identities of static properties.

A method was developed which made it possible to predict the environmental creep behavior for an arbitrary test condition during which overaging at 350°F took place and subsequently, when creep resistance was restored as the result of a recovery mechanism. The technique was based on the strain hardening rule compensated with empirically determined factors to account for creep damage and/or subsequent restoration of creep resistance.

During the examination of compatibility of mathematical forms for the representation of the creep phenomenon, an expression ($\epsilon_c = Kt^N$) provided a good fit in the primary creep region and a reasonably good approximation for part of the secondary stage of creep; an apparently unique correspondence between the values of K and N and the test variables, stress and temperature also was revealed.

WADD TR 61-25, OTS Release

January 1961

SUBJECT: CRITERIA FOR COMPARING THE EFFECTIVENESS OF DAMPING TREATMENTS
INVESTIGATOR: D. J. Mead
CONTRACT: AF 61(052)-332, University of Southampton, Hampshire England
ABSTRACT: In this report, expressions are derived for the response of simple vibrating systems, from which criteria have been deducted to indicate the effectiveness of a damping treatment in attenuating the response. The criteria include factors by which the treatment increases the mass and stiffness of the system, together with the loss factor increment. The response quantities considered include bending stresses, accelerations, inertia forces and sound transmission associated with simple vibrating plates under harmonic and random excitation. Coincidence sound transmission is also briefly considered. It is shown that whereas the mass and loss factor increase is always advantageous, a stiffness increase in some instances is detrimental.

As an example, three different commercial treatments are compared on the basis of some of the criteria. With low treatment weights, the treatment providing the highest loss factor is superior judged by each criterion, but at higher weights according to some criteria a treatment having lower stiffness, density and loss factor is more effective. The existence of optimum treatment weights for maximum effect upon the response is also shown by some criteria.

WADD TR 61-97, OTS Release

March 1961

SUBJECT: ON INFLUENCE FUNCTIONS IN THE THEORY OF FORCED VIBRATIONS OF MEMBRANES
INVESTIGATOR: I. Torbe, D. I. G. Jones
CONTRACT: AF 61(052)-332, University of Southampton, United Kingdom
ABSTRACT: In this report a general method is outlined for the calculation of the response of membranes with arbitrary boundaries to arbitrary loadings.

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WADD TR 61-97 (Continued)

It is assumed that, by projecting the area of the given membrane on the surface of an unbounded membrane and then applying the given loading to this projection, the application of a suitable load distribution around the boundary of the projection will enable us to satisfy the boundary conditions appropriate to the given membrane. An attempt to find the distribution in question leads to a logarithmically singular integral equation of an unusual type. A few solutions are outlined.

JOINING

WADC TR 59-531, OTS Release

May 1960

SUBJECT: DETERMINATION OF THE CAUSES OF WELD-METAL CRACKING IN HIGH-STRENGTH STEELS AND THE DEVELOPMENT OF HEAT-TREATABLE LOW-ALLOY-STEEL FILLER WIRES FOR USE WITH THE INERT-GAS-SHIELDED ARC-WELDING PROCESS

INVESTIGATOR: Herbert W. Mishler, Robert E. Monroe, Perry J. Rieppel

CONTRACT: AF 33(616)-5878, Battelle Memorial Institute

ABSTRACT: The work conducted under this contract was divided into four phases: three dealt with the study of the causes of weld-metal cracking in SAE 4340 steel and the fourth was concerned with the study of filler wires for producing welds heat treatable to ultimate-tensile-strength levels greater than 225,000 psi.

Freezing-cycle hot-tension studies were made on various heats of SAE 4340 steel containing various sulfur and phosphorus contents. Results indicated that the combined sulfur and phosphorus contents of SAE 4340 steel should be kept below 0.025 percent to prevent weld-metal hot cracking. An intergranular phase believed to be associated with hot cracking was found during light and electron microscopy of high-phosphorus SAE 4340 steels. This phase was tentatively identified as the iron-iron phosphide eutectic. Two new restrained weld-metal-cracking tests were developed that will give a quantitative measure of weld-metal-cracking resistance and which also will be inexpensive to conduct. Filler wires were developed which can produce weld metals capable of being heat treated to various strength levels in the range of 225,000 to 280,000 psi ultimate tensile strength.

WADC TR 60-607, OTS Release

December 1960

SUBJECT: FUNDAMENTAL STUDIES ON THE MECHANISM OF ULTRASONIC WELDING

INVESTIGATOR: W. J. Lewis, J. N. Antonevich, R. E. Monroe, P. J. Rieppel

CONTRACT: AF 33(616)-6268, Battelle Memorial Institute

ABSTRACT: The fundamental mechanisms of ultrasonic welding and the application of this process to the joining of various heat-resistant materials were investigated.

Previous fundamental studies which had shown the effects of time, temperature, and clamping force on ultrasonic welds were expanded to include studies of the shear force during welding. The results of these studies exhibited considerable scatter, but several trends appeared to be indicated.

Ultrasonic spot welds made in various combinations alloys generally confirmed the results of previous work with these alloys. Cracks were found at the edges of the spot welds in most of the material combinations studied. The effects of these cracks on weldment properties varied with the material. The presence of a reaction zone, apparently consisting of intermetallic compounds, was apparent in ultrasonic welds made between titanium and stainless steel. These findings indicate that ultrasonic welding is not suitable for the production of aircraft-quality spot welds in the heat-resistant and dissimilar metal combinations included in this program.

WADD TR 60-607, OTS Release (Continued)

General limitations on the use of this process in its current state of development can be established from the work conducted. It is apparent as a material's hardness increases, and the ratio of tensile strength to yield strength decreases, that weldability by the ultrasonic process decreases.

NON-DESTRUCTIVE TESTING

WADC TR 59-412, OTS Release

November 1959

SUBJECT: NONDESTRUCTIVE TESTS FOR CERAMIC, CERMET AND GRAPHITE MATERIALS

INVESTIGATOR: Julian H. Lauchner, Dwight G. Bennett, George L. Morgan

CONTRACT: AF 33(616)-5687, University of Illinois

ABSTRACT: As a direct result of the stringent requirements placed upon ceramic, cermet and graphite materials when contemplated for use as components in flight and space travel, the feasibility portion of this study was divided into three areas. External and internal defects, residual stress, and particle size measurements in polycrystalline bodies were investigated and their subsequent effect on static strength indicated.

Radiographic studies of polycrystalline ceramic bodies resulted in internal flaw detection of one or more percent of the total body cross section. Surface flaws were more reliably detected in nonporous bodies by fluorescent penetrant inspection, however, neither method was found to be applicable to relatively porous bodies. Static loading of specimens, non-destructively tested, indicated the extreme importance of surface structure and texture, whereas internal flaws were in many cases negligible in strength considerations.

The state of residual stress existent in a body was found to be a first order factor in predicting performance. Controlled residual stress developments in composite bodies formed the basis of the second phase of the study. Thermal expansion, elastic moduli, shape factors and heat treatment were employed in development and analysis of residual stress levels. Sandwich and concentric cylindrical type specimens were prepared and subsequently subjected to transverse loads. Measured apparent specimen strengths were analyzed in terms of residual body stress. Change in apparent specimen strength was found to be a direct function of the residual stress in the surface layer; however, certain limiting factors were found. In the balanced stress system, specimen strength decreased when internal stress, either compressive or tensile, exceeded some critical value characteristic of the body.

Thermal treatment studies indicated that body density, resultant from body forming techniques or firing schedules, was a measure of body maturity and general freedom from macroscopic defects. Within limited density ranges, increased density values were observed to result in increased flexural strengths.

Relative particle size analysis by x-ray back reflection techniques correlated with flexural strength of alumina bodies for the average particle ranges between one and ten microns.

COATINGS

CERAMICS

WADD TR 60-157, OTS Release

May 1960

SUBJECT: ULTRASONICS AND CERAMIC COATINGS
INVESTIGATOR: R. R. Whymark, W. E. Lawrie
CONTRACT: AF 33(616)-6396, Armour Research Foundation
ABSTRACT: This report describes investigations into the use of ultrasonic techniques to determine strength of ceramic-metal bonds and to locate defects in the bonds. The techniques evaluated have involved the use of both high frequencies (1-10 mc/s), low frequencies (14 kc/s) and the simultaneous use of high and low frequencies. Experiments at low signal frequencies indicate the average bond strength of flame-sprayed zirconium oxide to be about 1500 psi. Promising results have been obtained using high frequency transmitted energy and a Schlieren detection system. Apparatus is designed to generate surface acoustic waves. The application of surface waves to defect detection is considered. An experimental method has been developed to utilize intermodulation between high and low frequency sound waves to indicate bond continuity. Intermodulation techniques obviate the need for repeatable coupling of transducers which is required in direct high frequency transmission methods. Consideration is given to ultrasonic image converter systems.

WADD TR 60-317

July 1960

SUBJECT: RESEARCH ON CERAMIC COATINGS WITH CONTROLLED REFLECTIVE AND EMISSIVE PROPERTIES
INVESTIGATOR: D. G. Burgess, J. R. Jasperse, L. Marcus, W. S. Martin, E. P. Flint
CONTRACT: AF 33(616)-6371, Arthur D. Little, Inc.
ABSTRACT: Two types of ceramic coatings were formulated for application to Inconel in order to give: (a) the highest possible emissivity from room temperature to about 1200°C, and (b) the highest possible reflectivity to solar energy (0.4 to 2.0 microns) while having high emissivity in the infrared from 25° to 700°C or higher. Both types of coatings were formulated from a modified barium silicate frit. This frit was opacified with various black spinels to give the (a) type of enamels having total normal emittances of close to 0.8 at thicknesses of 5 mils or greater, with only minor changes over the specified temperature range. The (b) type enamels were made up with equal parts of stannic and ceric oxides as opacifiers. At thicknesses greater than 5 mils, these have hemispherical reflectances varying between 0.62 and 0.67 at wave lengths of 2.6 microns and 0.6 micron, respectively; together with total normal emittances of 0.75 at 400°C and 0.60 at 800°C.

WADD TR 60-427, OTS Release

October 1960

SUBJECT: INVESTIGATION OF THE FATIGUE PROPERTIES OF MOLYBDENUM UNDER VARIOUS CONDITIONS OF TEMPERATURE, COATINGS, AND STRESS CONCENTRATION
INVESTIGATOR: Alexander A. Mittenbergs, Dean N. Williams, Robert I. Jaffee, Horace J. Grover

WADC TR 53-373, Sup 8

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WADD TR 60-427, OTS Release (Continued)

CONTRACT: AF 33(616)-5915, Battelle Memorial Institute
ABSTRACT: Fatigue behavior of unalloyed arc-cast molybdenum was investigated in tension-tension fatigue loading on uncoated and coated, unnotched and notched sheet specimens at 1800°F, room temperature, and -40°F. Three oxidation-resistant coatings were evaluated under fatigue loading. Static tensile tests were also conducted on specimens of the four surface conditions at the three temperatures. The fatigue strength of unprotected molybdenum at 1800°F was in the same range as the fatigue strength of some other high-temperature materials at this temperature. At room and low temperatures, the fatigue strength of unprotected specimens was relatively high. The notch sensitivity of unprotected molybdenum was low at 1800°F, but rather high at room and at low temperatures. All coatings lowered the fatigue strength of molybdenum at all three temperatures. The loss of fatigue strength was higher in the notched specimens. The coatings investigated did not prove to be completely reliable for high-temperature applications under continuous fatigue loading. For short-life high-temperature fatigue loading, however, utilization of coated molybdenum appeared to be possible with proper precautions. Further research is suggested on protective coatings and on molybdenum-base alloys.

ORGANIC

WADC TR 55-58, Part VI

October 1960

SUBJECT: NUCLEAR RADIATION RESISTANT POLYMERS AND POLYMERIC COMPOUNDS
INVESTIGATOR: John W. Born
CONTRACT: AF 33(616)-6442, B. F. Goodrich Company
ABSTRACT: This research includes the following: fundamental studies of radiation energy transfer and of the mechanisms of radiation damage in high polymers; the selection, design, and synthesis of special new monomers to produce new high polymers with outstanding inherent radiation resistance and heat stability; polymerization of said monomers, identification of the resulting polymers, and evaluation of their radiation and heat stabilities; applied studies of the effect of gamma irradiation in air at room temperature on the compression set properties of various rubber compounds, with and without potential antirads present; and static and semi-dynamic radiation testing of "O" rings with attempts to protect the military-approved rubber compounds against radiation damage with antirads.

The work which is reported includes fundamental, basic, and applied research. The results are stated briefly in the summary which follows.

WADD TR 59-14

May 1960

SUBJECT: INVESTIGATION OF THE THERMAL STABILITY OF VARIOUS SLIGHTLY SOLUBLE SALTS AND THEIR PROPERTIES

WADC TR 53-373, Sup 8

WADD TR 59-14 (Continued)

INVESTIGATOR: Edward R. Allen, Julius Ciric
CONTRACT: AF 33(616)-6160, Rutgers University
ABSTRACT: To meet the ever increasing demand of high temperatures, corresponding inhibitive materials are needed. Of the array of materials suggesting themselves the chrome bearing zinc composition, zinc yellow, its cadmium counterpart, two basic chromates of zinc, the alkaline earth chromates, and the alkaline earth chromito-chromates were studied. The temperature range was restricted to the 1000°C zone.

Zinc yellow showed some deterioration in the 200-250°C range and a substantially complete breakdown between 300-400°C. This was shown by marked color change, appreciable losses in weight and in chromium (VI). The basic chromates of zinc were slightly superior to zinc yellow. The cadmium counterpart of zinc yellow was stable to 550°C-600°C.

The alkaline earth chromates appeared to be more promising. Calcium chromate showed a gradually increasing change from just above 600°C and reached an essentially complete breakdown, accompanies by fusion at 950°C. Strontium chromate showed little change when heated for 12 hours at 1000°C.

Both calcium and strontium chromate are sensitive to impurities, being most sensitive to their respective oxides. Upon calcination of these chromates with their corresponding oxides even more promising products are obtained, the chromito-chromates. The calcium derivatives show an increase in thermal stability with an increase in percentage of the oxide. The optimum composition, however, remains to be determined. Both the calcium and the strontium derivatives are stable for 12 hours at 1000°C. The former show a decrease in solubility over the unmodified calcium chromate, while the one composition of strontium derivative showed an increase in solubility as compared with the unmodified strontium chromate.

Extensive solubility studies were made on many of the materials under investigation. The indications are that this property may be an index of thermal stability.

The conclusion at this time is that the alkaline earth chromates and chromito-chromates deserve further study.

WADD TR 60-126

August 1960

SUBJECT: THE VACUUM-THERMAL STABILITY OF ORGANIC COATING MATERIALS
PART I. The Polyurethanes
INVESTIGATOR: James J. Mattice
ABSTRACT: This report is divided into two sections. Section 1 is a survey of the basic knowledge of polyurethane chemistry and of the research which has been conducted in studying the synthesis and degradation reactions of these materials. The application of this information in studying the adverse effects of the high vacuum of space and high temperature is emphasized.

WADD TR 60-126 (Continued)

Section 2 presents the results of the vacuum-thermal exposures of commercially available, unmodified polyurethane resins. The relationship between structure, cure, film thickness and weight losses of the polymers is discussed. The urethane bond appears to be the major labile species, leading to a characteristic degradation, regardless of structure which is complete at 500°F. The physical appearance and properties of degrading films is good and pigmentation of a film with titanium dioxide has different effects at differing temperature levels.

WADD TR 60-386

October 1960

SUBJECT: SPECTRALLY SELECTIVE COATINGS FOR TEMPERATURE CONTROL OF SPACE PROBES

INVESTIGATOR: Robert M. Van Vliet, James J. Mattice, Robert A. Cross

ABSTRACT: Two special purpose organic coatings were developed for evaluation on a short duration space probe. The coatings required very accurate tailoring of the reflective and emissive properties for temperature control of the space vehicle. The coatings were designed for solar absorptivities (α_s) of 0.53 and 0.94 with emittances of 0.95 and 0.8, respectively.

In order to make the first coating above, absorbing and reflecting pigments were used in combination. It was found that α_s varied with log of the absorbing pigment concentration when using a constant concentration of reflecting pigment. This derived relationship permitted exact control of the solar absorption. The emittance was controlled by selecting pigments that were absorbing in the infrared (even though they reflect short wavelength light).

The latter coating was required to be black in the visible and near infrared but gray in the far infrared. This was accomplished by using pigments that were highly absorbing at short wavelengths and partially transparent at long wavelengths. Aluminum flake was then added to this coating to give improved infrared reflectance (to form a gray) but without changing the short wavelength absorption.

Useful relationships were developed in carrying out these studies. Of greatest interest is the fact that the blending of black and white pigments appears to follow an exact mathematical relationship over a wide range of reflectivities. Also, the selective reduction of emittance of organic coatings with leafing aluminum pigment has been established even for highly pigmented enamels.

WADD TR 60-703, Part I

November 1960

SUBJECT: THE ULTRAVIOLET DEGRADATION OF ORGANIC COATINGS PART I.
Degradation in Air

INVESTIGATOR: A. L. Alexander, F. M. Noonan, J. E. Cowling, R. E. Kagarise

CONTRACT: MIPR 33(616)-59-21, U. S. Naval Research Laboratory

WADC TR 53-373, Sup 8

WADD TR 60-703, Part I (Continued)

ABSTRACT: A research program to develop suitable coatings for space vehicles, and for studying the effects of ultraviolet radiation on typical polymeric coatings in air is described.

The initial study under this program required irradiating a number of typical polymeric coatings with a near-ultraviolet source at ambient conditions for about 300 hours. It was observed that the relative stability of the polymeric materials which were irradiated roughly parallels their relative order of stability to gamma radiation. Infrared data indicate the relative stability, in decreasing order, to be as follows: polystyrene, melamine formaldehyde, urea formaldehyde, styrenated alkyd, soya oil alkyd, silicones, and polyvinyl chloride.

These initial experiments, qualitative and largely exploratory in nature, indicate that infrared spectroscopy affords a satisfactory means of qualitatively following the chemical changes induced by the ultraviolet.

WADD TR 60-703, Part II

November 1960

SUBJECT: THE ULTRAVIOLET DEGRADATION OF ORGANIC COATINGS PART II.
Degradation in Vacuum
INVESTIGATOR: A. L. Alexander, F. M. Noonan, J. E. Cowling, Suzanne Stokes
CONTRACT: U. S. Naval Research Laboratory
ABSTRACT: This report is a continuation of that described in Part I of this series which was largely concerned with the effects of ultraviolet radiation on polymers in the presence of a normal atmosphere. This work now has been extended to include studies of the effects of very short wavelength radiation (i.e., between 1150 and 2000 Angstroms) on polymeric films in a high vacuum. A comparison of the effects of ultraviolet radiation on several polymeric films in air with those obtained in absence of air (high vacuum) point to the possibility that some organic coatings may be much better able to withstand a space environment than normal weathering at the earth's surface. It appears that the dominant effect of ultraviolet radiation in space may be to cause crosslinking, while in the normal atmosphere at the earth's surface numerous volatile scission products are formed, usually as a result of reactions with oxygen and moisture of the atmosphere.

Experimentation of the type described in this and the previous report are continuing.

WADD TR 60-704

February 1961

SUBJECT: STUDIES ON THE PROTECTIVE ULTRAVIOLET ABSORBERS IN A HIGH VACUUM ENVIRONMENT. II
INVESTIGATOR: Richard G. Schmitt, Robert C. Hirt
CONTRACT: AF 33(616)-5945, American Cyanamid Co.

WADD TR 60-704 (Continued)

ABSTRACT: Compounds which are effective in protecting materials from terrestrial ultraviolet radiation may, in a space environment, suffer loss both by evaporation and by photochemical degradation from the shorter wavelength ultraviolet radiation. The loss of absorber by evaporation from plastics was found to be negligible for thermosetting resins such as melamine-formaldehyde and polyesters. Evaporation from thermoplastic resins such as cellulose acetate and methyl methacrylate was appreciable for the more volatile absorbers. The photochemical stability of the ultraviolet absorbers has been studied in air and in vacuum in the 2000-4000 Angstrom region using an AH-6 high pressure mercury arc to simulate sunlight above the earth's atmosphere. It was determined that the ultraviolet absorbers are considerably less stable in a space environment and that the loss in stability is due primarily to a higher quantum yield for the short wavelength ultraviolet radiation. A rapid decrease in the rate of decomposition was observed for the absorbers dispersed in polymeric substrates; this was attributed to a decrease in the rate of energy absorption due to the formation of decomposition products of both absorber and polymer. The stability of the organic absorbers can be improved by the use of certain stable ferrocene derivatives. The effectiveness of the ultraviolet absorbers in protecting polyester resins from photochemical degradation under terrestrial and extraterrestrial conditions was studied. The absorbers were found to be much less effective in a space environment due to the strong ultraviolet absorption of the polyester at the shorter wavelength which competes for the incident radiation. This latter effect, the lower effectiveness of the absorbers, is a more important factor than the decrease in photochemical stability for strongly absorbing polymers. At the present time, the use of more transparent polymers containing one or more absorbers is recommended for long exposures to a space environment.

CORROSION

WADD TR 60-384, OTS Release

July 1960

SUBJECT: NITROGEN TETROXIDE CORROSION STUDIES
INVESTIGATOR: C. W. Alley, A. W. Hayford, H. F. Scott, Jr.
CONTRACT: AF 33(616)-6568, Nitrogen Division, Allied Chemical Corp.
ABSTRACT: The purpose of this investigation was to determine quantitatively the corrosive effects of nitrogen tetroxide on mild steel, aluminum, stainless steels and titanium. This was done under static conditions at six water concentrations up to 3.2 wt % and four temperatures up to 74°C. The corrosion rates under dynamic flow conditions were also investigated.

The corrosion of carbon steel (ASTM A-285 Grade C) and aluminum (5086) was less than 0.5 mil per year in nitrogen tetroxide containing up to 0.2 wt % water at 74°C, increasing to 50 mils per year at 3.2 wt % water and 74°C. Negligible corrosion was observed under severe conditions with stainless steel (304-L) and titanium (75A and 6Al-4V) whereas high strength steel (PH 15-7 Mo) showed losses of 0.5 to 1.0 mils per year. No stress corrosion cracking was observed in tests of carbon steel, high strength steel or aluminum in nitrogen tetroxide containing 0.1 and 1.6 wt % water at 49°C. Significant corrosion of stainless steel (304-L) occurred in the presence of Teflon.

Dynamic tests showed no significant corrosion of 304-L and PH 15-7 Mo stainless steels and average rates of 0.05 mils per year for aluminum and 0.33 mils per year for carbon steel after 205 hours exposure to commercial nitrogen tetroxide flowing at velocity of 10 ft per second at 30°C.

WADD TR 60-436

August 1960

SUBJECT: THE COMPATIBILITY OF VARIOUS METALS AND CARBON WITH LIQUID FLUORINE
INVESTIGATOR: Charles J. Sterner, Alan H. Singleton
CONTRACT: AF 33(616)-6515, Air Products, Incorporated
ABSTRACT: Experimental studies were made to determine the compatibility and resistance to corrosion of various metals and carbon with liquid fluorine at -320°F. The metals tested were various alloys of aluminum, stainless steel including high-strength steels, titanium, copper, Monel metal, nickel, and magnesium. Tests which were performed included: continuous immersion of stressed and unstressed samples in liquid fluorine for periods up to two weeks; impact ignition of titanium and aluminum in liquid fluorine and of titanium in liquid oxygen at impact energy levels ranging from 2.6 to 65 ft-lb; intensive and extensive impact on tubes containing liquid fluorine; passivation and storage for periods up to 64 days followed by immersion in liquid fluorine; thermal shock of samples, both by liquid fluorine and in contact with liquid fluorine; flexing of metal samples immersed in liquid fluorine; testing of metal samples while immersed in liquid fluorine; the explosibility of contaminant in liquid fluorine.

It was found that the corrosion of the metals tested in pure liquid fluorine was negligible, generally amounting to less than 1 mil penetration per year. However, contamination of liquid fluorine can result in severe

WADD TR 60-436 (Continued)

corrosion. Graphitic carbon was found to be incompatible although dense amorphous carbon was affected only slightly. Titanium was found to ignite upon impact in liquid fluorine although the ignition did not propagate. No evidence was found to support the theory that a fluoride film is required to protect metals from attack by liquid fluorine.

Passivation by gaseous fluorine is recommended as an extension of the cleaning procedure despite the lack of real evidence that passivation is required for materials which have been thoroughly cleaned.

POLYMER AND SYNTHESIS STUDIES

WADC TR 57-126, Part IV

August 1960

SUBJECT: PHOSPHINOBORINE POLYMERS
INVESTIGATOR: Ross I. Wagner
CONTRACT: AF 33(616)-5435, American Potash & Chemical Corp.
ABSTRACT: A series of preparations of linear dimethyl-, methylethyl- and diethylphosphinoborine polymers have been made to optimize experimental conditions. N-Methylpiperazine when used as an end-group for linear dimethylphosphinoborine polymers provided a fair yield of product which, except for a large melting range, resembled polymers terminated with other end-groups. Triethylenediamine markedly reduced the rate of dimethylphosphine borine pyrolysis and failed to yield a linear polymeric product. Qualitative solubility data have been obtained for the linear methylethyl- and diethyl- derivatives. Attempts to prepare linear phosphinoborine polymers either by pyrolysis of trimethylphosphine trichloroborine or dimethylphosphinoborine trimer with triethylamine or by alkylation of methylphosphinodimethylborine polymer were unsuccessful. Linear methylethylphosphinoborine polymer was only partially chlorinated on treatment with methyl chloride--aluminum chloride in contrast to the dimethyl-derivative. A further attempt to open the dimethylphosphinoborine trimer ring by heating with triethylenediamine resulted instead in a polymer formed by opening the ring structure of the diamine.

Dimethylphosphinoborine trimer was prepared in good yield from dimethylphosphonium chloride and lithium boro hydride and in low yield by dehydrohalogenation of dimethylphosphine chloroborine. Dehydrohalogenation of dimethylphosphine diethylchloroborine and dimethylphosphine trichloroborine failed to give cyclic trimeric products but the latter appeared to yield low molecular weight linear phosphinoborine polymers. The stoichiometry of the fluorination of dimethylphosphinoborine trimer has been established experimentally but no success has been achieved in attempts to cyanogenate the trimer. An X-ray diffraction study of the dimethylphosphinoborine--methylphosphinoborine pentamer indicated the crystals to be of complex structure. Dimethylphosphinodichloroborine trimer appears to be less thermally stable than the parent compound.

The synthesis of phosphine intermediates by known methods has produced one new secondary phosphine and one new organophosphine derivative. Nuclear magnetic resonance spectra of the phosphine, C_2H_5P , prove it to be cycloethylenephosphine rather than the isomeric vinylphosphine.

N-Dimethyl-B-trimethylborazene has been synthesized both by dehydrohalogenation of a mixture of the methyldibromoborine adducts of methylamine and ammonia and by pyrolysis of a mixture of the trimethylborine adducts of methylamine and ammonia. The isomeric N-trimethyl-B-dimethylborazene was prepared by reaction of N-trimethylborazene with a deficiency of methylmagnesium bromide. The attempt to prepare decamethylbiborazyl by pyrolytic dehydrogenation of the two isomeric pentamethylborazenes resulted in formation of by-product methane and a polymeric borazene derivative. The pseudoaromatic 8-bora-7,9-diazaro-peri-naphthene has been prepared and studied briefly.

WADC TR 57-126, Part IV (Continued)

Dimethyl- and diphenylthiosiloxane polymers have been prepared by reaction of the corresponding dichlorosilanes with hydrogen sulfide. In the presence of trimethylchlorosilane the preparation of diphenylthiosiloxane polymers resulted in a high yield of a single crystalline solid, presumably the trimer or dimer.

WADC TR 58-51, Part III

April 1960

SUBJECT: HIGH POLYMERIC MATERIALS
INVESTIGATOR: C. S. Marvel, L. F. Audrieth, T. Moeller, J. C. Balar, Jr.
CONTRACT: AF 33(616)-5486, University of Illinois
ABSTRACT: C. S. MARVEL SECTION

The work described herein is in part a continuation of that reported in WADC TR 58-51 Pt II.

Further work on p-polyphenyl has been initiated. The chloranil dehydrogenation of poly-1,5-cyclohexadiene has been found to involve a complex interaction of the two materials rather than simple dehydrogenation.

The cyclic intramolecular-intermolecular polymerization of diphenyl derivatives of 1,5-hexadiene, 1,6-heptadiene and 1,7-octadiene has been investigated. While new polymers with recurring cyclic units have been obtained, their heat stabilities do not appear to be outstanding.

A new type polymer which contains recurring benzene and thiazole rings has been prepared and preliminary studies show these to be remarkably stable at 300°C. Some polymers with good inherent viscosities have been prepared but they have not yet been obtained in other than powder form.

Other work on ethylene-anthracene copolymers, attempts to make polyperhydronaphthalene and attempts to make a ladder type polymer with alternating benzene and pyridine rings is recorded.

L. F. AUDRIETH - T. MOELLER SECTION

The substitution of two chlorine atoms in trimeric phosphonitrilic chloride has been effected by treatment in acetone with ammonium thiocyanate in appropriate stoichiometry. The resulting compound polymerizes to a rubber-like product at 170°C. A similar reaction in acetonitrile involving tetrachloro-diphenyl-triphosphonitrile yields dichloro-diphenyl disothiocyanato-triphosphonitrile. Tetrameric phosphonitrilic chloride in acetone yields an oil with the analytical composition $P_4N_4Cl_6(NCS)_2$, which can be characterized through its ethanol and aniline addition products, but the pure compound has not been isolated.

A general method for preparing di-aminolysis products of tetrameric phosphonitrilic chloride, $P_4N_4Cl_6(NHR)_2$ or $P_4N_4Cl_6(NR_2)_2$, by treating the chloride in ether with either the free amine or with an aqueous

solution of the amine hydrochloride and sufficient potassium hydroxide to maintain neutrality has been developed. The two-phase reaction is best applied to strongly basic aliphatic amines. Direct reaction under anhydrous conditions is more useful for the more weakly basic aromatic amines. Several isomers have been prepared.

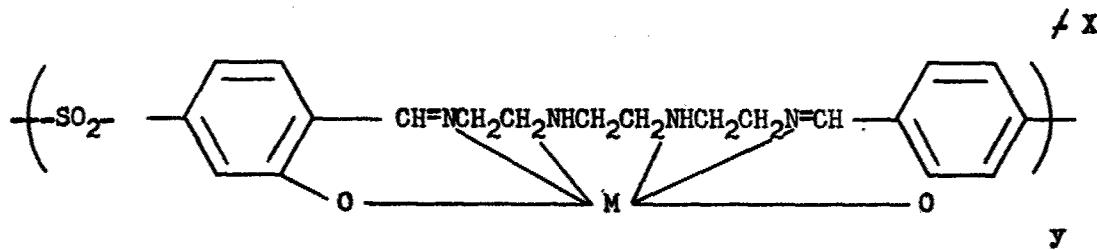
Complete aminolysis of trimeric phosphonitrilic chloride has been effected with a series of lower aliphatic monoamines and with ethylenediamine. Higher diamines appear to yield polymeric products.

Some indication of exchange of halogen between trimeric phosphonitrilic chloride and potassium bromide has been obtained.

J. C. RAILAR, JR. SECTION

Efforts to form linear polymers by linking organic groups together by coordination with metal have continued. A few of the substances which have been formed show good heat stability. In no case has a polymer of high molecular weight been obtained. Efforts are under way to modify reaction conditions so as to favor the formation of longer and more stable chains.

Because multiple chelation often increases stability, Dr. Goodwin has been preparing polymers of the type

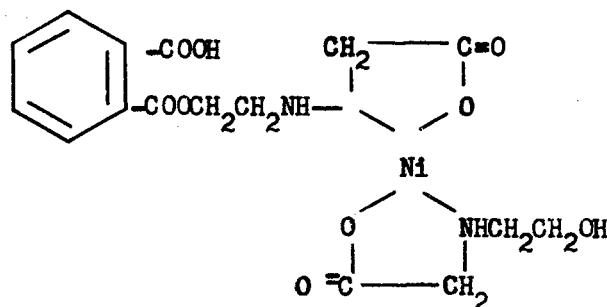


where M represents an atom of metal with a coordination number of 6, and X depends upon the oxidation state of M.

Dr. McLean has prepared compounds of bis-salicylic acids and bis-diphenols, in which the coordinating ion is either that of a metal or is silicon. Polymeric substances have been obtained (some of them of good stability) but their composition is indefinite. Dr. McLean left us in August, and Mr. Fujikawa is continuing the work, using aliphatic dihydric alcohols, such as pinacols and pentaerythritol.

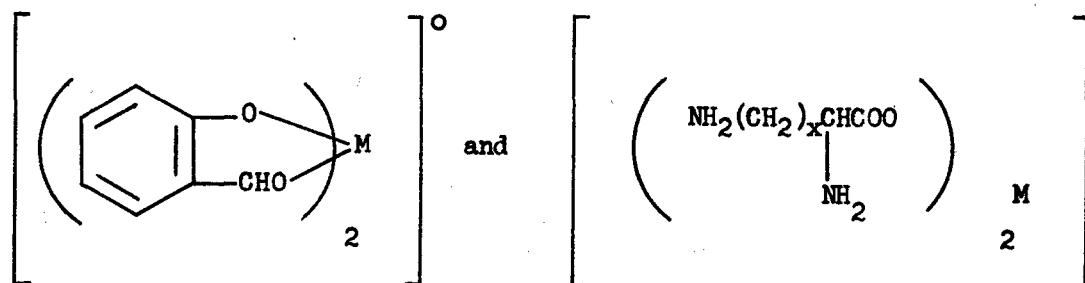
WADC TR 58-51, Part III (Continued)

Mrs. Moraghan and Mrs. Chen have reviewed some of our earlier work in the hope of making improvements. It was shown earlier that compounds of the type



can be made to "self-condense" to form short-chain polymers. Mrs. Moraghan is trying to find conditions that will allow increase in chain length.

Mrs. Chen spent some time on a study of increasing the chain length of the polymers formed by the reaction between



She was able to get substances which evidently contain between twenty and thirty atoms of metal. However, they lack stability.

WADC TR 58-187, Part III

May 1960

SUBJECT: SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE STAPLE AND NUCLEAR RADIATION STAPLE METAL-CYCLOPENTADIENYL FLUIDS

INVESTIGATOR: Robert L. Schaaf, Peter T. Kan, Kenneth L. Rinehart, Jr.

CONTRACT: AF 33(616)-5053, Wyandotte Chemicals Corp., and University of Illinois

ABSTRACT: In a search for liquids suitable for use as high-temperature hydraulic fluids and lubricants, six new siloxanylferrocenes were synthesized, and the thermal stability, viscosity and fluid range were determined. Efforts to find feasible routes to 1,1'-bis(aryloxyphenyl)-ferrocenes, polychloroferrocenes, and polymeric perfluoroalkylbenzimidazoles were unsuccessful.

WADC TR 58-187, Part III (Continued)

From the lithiation and subsequent carbonation of dimethyl-ferrocene, the enantiomorphic forms of 1,1'-dimethylferrocene-3-carboxylic acid were isolated, and 1,1'-dimethylferrocene-2-carboxylic acid and a mixture of di-acids were obtained. By reduction of the required esters with lithium aluminum hydride, mono- and bis-hydroxymethylferrocenes were prepared. Oxidation of desoxyferrocene, methyl-, and ethyl-ferrocene with manganese dioxide gave ferrocil, formyl-, and acetyl-ferrocene, respectively. The reduction of nitroferrocene, prepared from n-propyl nitrate and lithioferrocene, was investigated. Unsuccessful were attempts to prepare nitrosoferrocene, 1,1'-diamino-ferrocene, and to unite hydroxymethylferrocene with methyl linolenate or ethyl linoleate.

WADC TR 58-382, Part II

May 1960

SUBJECT: SYNTHESIS AND EVALUATION OF NEW POLYMERS PREPARED BY
STEREOSPECIFIC CATALYSIS
INVESTIGATOR: Charles G. Overberger
CONTRACT: AF 33(616)-5253, Polytechnic Institute of Brooklyn
ABSTRACT: 3-Trifluoromethyl-1-butene and 4,4,4,-trifluoro-1-butene
have been prepared. Trifluoromethylacrylonitrile was synthesized and polymerized with several bases.

Methylenecyclobutane of high purity was obtained from 1,1-bis-(hydroxymethyl)-cyclobutane. Vinylcyclopropane of high purity was obtained from 1-cyclopropylethyl-S-methylxanthate while the pyrolysis of 1-cyclo-propylethyl acetate yielded cis 1,3-pentadiene as the major product. Cyclo-butylethyl acetate was prepared and pyrolysis of cyclobutylethyl acetate yielded a mixture of olefins. Cyclobutanecarboxaldehyde was synthesized and found to trimerize to a cyclic ether which has been characterized. A lactone reduction product of 4-acetyl-3,3-dimethylcyclobutylacetic acid has been characterized and reduced to a diol which has also been characterized. A two step reduction of 4-acetyl-3,3-dimethylcyclobutylacetic acid yielded a cyclo-butylethanol, which has been characterized, and an unidentified ether. 3-Cyclo-hexenylmethyl acetate was prepared, and the pyrolysis of this ester yielded a mixture of methylenecyclohexene, toluene, methylenecyclohexane and several additional unidentified olefins. Methylenecyclohexene of high purity was obtained from 3-cyclohexenylmethyl-S-methylxanthate. Pure cis or trans 2-methyl-vinylcyclohexane has been synthesized, characterized and polymerized to an insoluble polymer with a triisobutylaluminum-titanium tetrachloride catalyst.

An isotope effect was not observed in the polymerization of alphadeuterovinylcyclohexane in a comparison with the polymerization of vinylcyclohexane with a triethylaluminum-titanium tetrachloride catalyst.

The DP of polymethacrylonitrile from potassium in ammonia catalysis was approximately independent of both monomer and catalyst concentrations. Potassium amide catalysis produced an acetone insoluble polymer, and potassium hydroxide polymerized methacrylonitrile in ammonia. Water exerted an observable effect on potassium or potassium amide catalysis in the polymerization of methacrylonitrile in ammonia.

SUBJECT: SYNTHESIS AND EVALUATION OF NEW POLYMERS PREPARED BY STEREOSPECIFIC CATALYSIS

INVESTIGATOR: Charles G. Overberger

CONTRACT: AF 33(616)-5253, Polytechnic Institute of Brooklyn

ABSTRACT: Cyclopentane was identified as the major product from the pyrolysis of 1-cyclopropylethyl acetate. Polyvinylcyclopropane was prepared, and the polymer modified by ring opening with hydrogen bromide, bromine, p-toluenesulfonic acid, and acetic-sulfuric acid mixture. 2-Trifluoromethyl-propyl bromide was prepared and used to alkylate ethyl malonate. 3-Trifluoromethylbutene-1 was prepared by pyrolysis of the primary acetate. Ethyl 3,3-methylcyanobutyrate was prepared but could not be reduced to the corresponding cyano-alcohol. 1,1,1-Trichloro-3-bromopropane was converted to 1,1,1-trifluoro-3-bromopropane. Basic hydrolysis of 4,4-methylcyanopentene-1 yielded 2,2-dimethylpent-4-enoic acid. 2-Trifluoropropane was prepared, but attempts at alkylation were not successful. 6,6,6-Trifluorohexene-1 was prepared but could not be obtained in a pure state. Homogeneous polymerization of trifluoromethylacrylonitrile was effected. A methoxymercuriacetate derivative of 2-trifluoromethyl propene was prepared. A mixture of methylenecyclobutane, spiropentane, and 2-methylbutene-1 was the product of the reduction of pentaerythrityl tetrabromide with zinc in various solvents. Methylenecyclobutane was purified by distillation, and also by regeneration from a silver nitrate solution. Methoxymercuration of methylenecyclobutane gave a solid derivative from which the monomer could be regenerated. The yield of polymethylenecyclobutane from polymerization with titanium tetrachloride-aluminum triisobutyl was found to increase with increasing temperature. Acid catalyzed rearrangement to 1,1-bis-(hydroxymethyl) cyclopropane gave cyclobutanecarboxaldehyde. The corresponding cyclobutane gave cyclopantanecarboxaldehyde. It was found that the solvent strongly influences the reactivities of isobutylene, p-chlorostyrene when copolymerized with aluminum bromide or stannic chloride. The molecular weights of polystyrene polymerized in solvents of varying dielectric constants with aluminum bromide or boron trifluoride etherate as catalyst were determined. The xanthates of 1-cyclobutyl-ethanol and 3-cyclohexenylmethanol were prepared. Methyl vinyl sulfone was prepared and a high vacuum manifold was constructed for its polymerization.

SUBJECT: RESEARCH ON POLYMERIC BONDING SYSTEMS AND THEIR DIELECTRIC BEHAVIOR

INVESTIGATOR: R. P. Anderson, M. M. Sprung

CONTRACT: AF 33(616)-5535, General Electric Research Laboratory

ABSTRACT: Further work on the formation of borosiloxane polymers, particularly by the reaction between an acetoxysilane and an alkoxyborane or by the reaction between an alkoxy silane and an acetoxyborane, is described in this report. A variety of low polymers, bearing different substituents on silicon and on boron, were prepared. Several nonlinear, hydrocarbon-insoluble fractions were isolated, and it was shown that disproportionation sometimes, but not necessarily always, occurs. Some dephenylation of a phenylborane moiety almost always occurs when high temperatures are employed.

WADC TR 59-61, Part II (Continued)

In the reaction of an aminoalkylalkoxysilane with boron acetate, the amino substituent enters the reaction by elimination of acetic acid. A primary amine will undergo group interchange with an acetoxy silane.

Dichloro-p-(dimethylaminophenyl)methylsilane and dichloro-p-(dimethylaminophenyl)phenylsilane were prepared and converted to the corresponding diacetoxy silanes. Dichloro-p-(dimethylaminophenyl)phenylsilane was hydrolyzed nearly quantitatively to the corresponding diol, and the diol was reacted with benzeneboronic acid to produce a nitrogen-substituted borosiloxane.

The equilibrium of cyclic siloxanes and boroxanes were studied briefly. Here dephenylation is negligible at reasonable temperatures, but oxidation is still possible. Thermal equilibration, with the boron component acting as an auto-catalyst, is a slow process but gives better results than base catalyzed equilibration.

Several amine complexes of tributyl borate were prepared. Crystalline complexes of triphenylboroxole and amines were not obtained, but interesting, stable "adducts" were obtained with several polyethylene amines. A similar adduct was obtained from poly(triphenylborosiloxane) and tetraethylene pentamine.

Several aminoalkyl borates were prepared and their properties compared to simple alkyl borates. These data were then extended to linear polymers formed from alkyl borates with diols or aminodiols; and to crosslinked polymers formed from alkyl borates with diols, oxadiols, or aminodiols. It was observed that whenever five-membered rings can be formed through B-N dative bonding, this reaction route was favored almost exclusively over polymer formation.

WADC TR 59-64, Part II

March 1960

SUBJECT: THERMAL DEGRADATION OF POLYMERS UP TO 1200°C
INVESTIGATOR: Samuel L. Madorsky & Sidney Straus
CONTRACT: AF 33(616)-58-8, National Bureau of Standards
ABSTRACT: Samples of three thermoset plastics, Vibrin, epoxy, and phenolic, and of three high-temperature polymers, poly(vinylidene fluoride), polytrivinylbenzene, and polyacrylonitrile, were pyrolyzed in a vacuum at 1200°C. The relative amounts of gaseous, liquid, wax-like, and solid products were determined. The more volatile products were analyzed in a mass spectrometer, while the less volatile ones were tested for their overall average molecular weight by microcryoscopy. Chemical analysis was made of the original materials, and also of the residues from pyrolysis at 500, 800, and 1200°C in the case of phenolic resin, poly(vinylidene fluoride), polytrivinylbenzene, and polyacrylonitrile. Pyrolysis of a high molecular weight linear polymethylene was conducted at 500, 800, and 1200°C in a vacuum and in helium, and the volatile products were analyzed by mass spectrometry and cryoscopy. A number of copolymers of styrene with di- or trivinylbenzene in various proportions were pyrolyzed in a vacuum, in the temperature range 346 to 456°C.

WADC TR 59-64, Part II (Continued)

Measurements were made of rates of thermal degradation, in a vacuum at various temperatures, of all the polymers mentioned above, except polymethylene. The corresponding activation energies calculated from the rates were 36, 51, and 18 kcal/mole, for Vibrin, epoxy, and phenolic resins, respectively, and 48, 31, and 73 kcal/mole for poly(vinylidene fluoride), polyacrylonitrile, and polytrivinylbenzene, respectively.

WADC TR 59-95, Part II

June 1960

SUBJECT: SYNTHESIS OF 1000°F STABLE BASE FLUIDS
INVESTIGATOR: James W. Dale, Iral B. Johns, Elizabeth A. McElhill,
John O. Smith
CONTRACT: AF 33(616)-5553, Monsanto Chemical Co.
ABSTRACT: The results of tests for thermal stability directed at obtaining thermally-stable fluids which are liquid at room temperature and usable to 1000°F are presented. In this extension of the project an additional 50 compounds were tested in the vapor phase, or the liquid or condensed phase using four different types of apparatus. Many of the compounds were synthesized.

A number of fluoroorganic compounds were prepared for thermal stability evaluation. Several perfluorocyclic compounds were prepared by fluorination of the corresponding aromatics, and in the case of $(CNF)_3$, by fluorination of $(CNCl)_3$ with SbF_3Cl_2 . Other fluoroorganics, phenyl- or pyridyl-substituted perfluoroalkanes, were synthesized from SF_4 and the corresponding ketones or carboxylic acids. Evidence was obtained of a new reaction of SF_4 with aryl oxalates and other oxalic acid derivatives.

Compounds found stable over 1000°F included dibenzothiophene tested in the condensed phase, and tetrakis (perfluoromethyl) pyrazine and perfluorocyclobutane-perfluoroisobutylene tested in the vapor phase.

WADC TR 59-129, Part II

July 1960

SUBJECT: MECHANISM OF CROSS-LINKING IN THE VULCANIZATION OF NEVÉLASTOMERIC POLYMERS
INVESTIGATOR: Kay L. Paciorek, Lawrence C. Mitchell
CONTRACT: AF 33(616)-5642, Wyandotte Chemicals Corp.
ABSTRACT: The objective of this program was the elucidation of the mechanism of amine cross-linking of fluoroelastomers. Two approaches were undertaken solution studies of the copolymers of vinylidene fluoride-chlorotrifluoroethylene (VF-CTFE) and vinylidene fluoride-perfluoropropene (VF-PFP), and investigations of certain model systems.

Solution studies revealed a great difference in the behavior of the two fluoroelastomers. Isolation of amine hydrohalides from room-temperature reactions of amines with fluoroelastomers in conjunction with the presence of unsaturation in the resulting polymers (shown by infrared examination), proved dehydrohalogenation to be the first step in amine induced cross-linking. Various mechanisms of cross-linking were postulated.

WADC TR 59-129, Part II (Continued)

Several syntheses of model compounds were proposed. A synthesis utilizing 1,1,5-trihydroperfluoropentanol resulted in models suitable for amine studies. Parallel syntheses were also initiated. Preliminary model compound studies utilizing 1,1,3,3,5,5-hexafluoro-1,5-hexafluoro-1,5-dibromo-pentane supported the findings of the solution studies.

WADC TR 59-190, Part II

May 1960

SUBJECT: SYNTHESIS OF THERMALLY STABLE EPOXIDE RESINS FOR DIELECTRIC APPLICATIONS
INVESTIGATOR: Lee M. Kindley
CONTRACT: AF 33(616)-5518, Melpar, Inc.
ABSTRACT: The objective of the program was to prepare organophosphorus epoxides capable of being cured to thermally stable resins. To achieve this objective, several synthesis routes were explored.

Three new tertiary phosphine oxides were prepared and characterized. These compounds and several known organophosphorus compounds, to be used as intermediates, were prepared and analyzed.

The characterization of new phenolic tertiary phosphine oxides, which were to be reacted with epichlorohydrin to yield phosphorus epoxides, proved to be a difficult problem. After considerable development work, results indicated that the problem was one of analysis rather than preparation.

The preparation of a phosphorus-containing epoxide resin by condensing tris (hydroxymethyl) phosphine oxide with an epihalohydrin was investigated in some detail. Phosphorus-containing resins having a small amount of oxirane oxygen were isolated from some of the reactions. Attempts were made to cure these products with the hope of obtaining a useful product, but only thermoplastic or very viscous liquids were obtained.

WADC TR 59-272, Part II

June 1960

SUBJECT: SYNTHESIS OF SEMI-INORGANIC FLUORINE POLYMERS
INVESTIGATOR: Henry C. Brown
CONTRACT: AF 33(616)-5616, University of Florida
ABSTRACT: The mechanism of the deammonation reaction of perfluoroalkyl amidines have been explored and the first step shown to be the formation of perfluoroalkyl imidines. These intermediate compounds readily form copper (II) and zinc chelates. Further steps in the deammonation reaction, which results finally in formation of a sym. triazine ring, are proposed.

The possible formation of a linear polymeric structure from the low temperature reaction of perfluoroglutaronitrile with perfluorobutyramidine is described. This structure is a possible precursor to a linear perfluoroalkyl-triazine polymer.

Copolymerization of perfluoroglutaronitrile with perfluorobutyronitrile in the presence of basic catalysts other than ammonia is described. Copolymerization of perfluoroglutaronitrile with perfluoropropionitrile and perfluoroacetonitrile in the presence of catalytic amounts of ammonia appears to furnish copolymers of physical characteristics different from those previously reported but with high temperature stability.

Cotrimeration of perfluoroacetonitrile with benzonitrile is demonstrated and the resulting compounds, 2,4-bis phenyl-6-trifluoromethyl-1,3,5-triazine and 2-phenyl-4,6-bis trifluoromethyl-1,3,5-triazine are considered as possible monomers.

WADC TR 59-345

May 1960

SUBJECT: HIGH-TEMPERATURE SYNTHESES OF NEW, THERMALLY-STABLE CHEMICAL COMPOUNDS

INVESTIGATOR: Russell C. Phillips, David L. Chamberlain, F. Alan Ferguson

CONTRACT: AF 33(616)-5940, Stanford Research Institute

ABSTRACT: Studies of high-temperature reactions for synthesis of new, thermally-stable chemical compounds or precursors to such compounds, are described. The first part of the program was a survey of the literature for high-temperature reactions (above 500°C) which might result in such compounds. On the basis of this initial survey, and on information from continuing literature surveys, the following three studies were undertaken:

1. Synthesis of difluoroacetylene as a precursor to thermally stable, perfluorinated aliphatic or aromatic polymers.
2. Synthesis of stable organometallic compounds using arcs to ionize the metal constituent.
3. Synthesis of thermally-stable thiophosphonitrilic compounds.

Of three different approaches conceived for the synthesis of difluoroacetylene, only pyrolysis of the sodium salt of tetrafluorosuccinic acid appears to have yielded the desired product, although the analytical findings are not yet conclusive. Investigations of the reaction between atomic fluorine and graphite and of the pyrolysis of difluoromaleic anhydride are not yet completed.

The reactions between metals ionized from arc electrodes, and organic liquids circulated past the arc, have failed thus far to yield measurable quantities of organometallic compounds.

Reactions between vaporized phosphorus sesquisulfide (P_4S_3) and nitrogen in an electric arc have yielded several amorphous compounds containing sulfur, phosphorus and nitrogen. One such compound was heated to over 700°C in argon with no visible signs of decomposition.

WADC TR 59-345, Part II

June 1960

SUBJECT: HIGH-TEMPERATURE SYNTHESIS OF NEW, THERMALLY-STABLE CHEMICAL COMPOUNDS

INVESTIGATOR: Lars C. Bratt, David L. Chamberlain, F. Alan Ferguson

CONTRACT: AF 33(616)-5940, Stanford Research Institute

ABSTRACT: Results of the continuing investigation of high-temperature reactions for the synthesis of new thermally-stable compounds is reported. The solid product obtained from the pyrolysis of disodium tetrafluorosuccinate is believed to possibly be a low molecular-weight polymer of difluoroacetylene whose presence, in addition to other carbon-fluorine groups, was indicated by mass spectral analysis of the volatile products.

Reaction of phosphorus sulfide (P_4S_3 or P_4S_{10}) vapors with nitrogen in an electric arc yielded inert, amorphous, thermally-stable solids containing sulfur, phosphorus and nitrogen. These as yet uncharacterized products were found to be insoluble in common organic solvents but hydrolyzed very slowly when extracted with boiling water.

The possibility of forming organic iron compounds by shock chilling a plasma-jet containing thermally-activated iron with benzene, dicyclopentadiene and cyclopentadiene was investigated. However, only an insignificant concentration of iron was found in any of the liquid products obtained from such reactions, indicating the absence of any organoiron derivatives.

WADC TR 60-282, Part I

May 1960

SUBJECT: DETERMINATION OF THE RELATIONS BETWEEN STRUCTURE AND RADIATION STABILITY OF ARYL ETHER FLUIDS

INVESTIGATOR: Josef J. Schmidt-Collerus, George E. Bohner

CONTRACT: AF 33(616)-5317, University of Denver

ABSTRACT: The polyphenyl ethers are of interest to the Air Force as potential lubricants because of their demonstrated resistance to thermal and radiolytic degradation. A study of the degradation mechanisms involved in the radiolytic breakdown of diphenyl ether and higher homologs has been initiated during the current research period. A better understanding of the degradation mechanisms involved in the radiolysis of these ethers should permit a more realistic evaluation of the performance of these ethers in actual use.

It has been found that the radiolysis of diphenyl ether produces small amounts of gaseous products, mostly hydrogen, and other compounds such as benzene, phenol, p-phenylphenol, other phenolic materials, higher boiling three and four-phenylethers, and rather high molecular weight polymeric material. Although the research has not yet clearly defined a degradation mechanism for the diphenyl ether, certain postulations have been made which can plausibly explain the observed experimental data. Further research must be done in order to clearly elucidate the degradation mechanisms.

WADD TR 60-283

June 1960

SUBJECT: EVALUATION OF EXPERIMENTAL POLYMERS
INVESTIGATOR: Charles D. Doyle
CONTRACT: AF 33(616)-5576, General Electric Co.
ABSTRACT: Thermogravimetric analysis (TGA) in dry N₂ is considered in detail as a method for comparing the intrinsic thermal stabilities of experimental polymers on both empirical and fundamental grounds. Two procedural decomposition temperatures are defined and discussed. One, the "differential procedural decomposition temperature" (dpdt), is based on the locations of recognizable curve features, while the second, the "integral procedural decomposition temperature" (ipdt), is based on areas under the curve. Kinetic analysis of volatilization data is discussed on the basis of both the Arrhenius rate equation and its integral. Two quasi-kinetic methods are discussed, one based on empirical time-temperature superposition; the other, on an empirical relationship between isothermal times and temperatures in TGA. Two corroborative test methods, differential thermal analysis and thermoparticulate analysis are discussed briefly.

WADD TR 60-315

June 1960

SUBJECT: SYNTHESIS OF NITROGEN-CONTAINING HETEROCYCLIC FLUID SYSTEMS
INVESTIGATOR: E. H. Kober, W. J. Schnabel, H. A. Schroeder
CONTRACT: AF 33(616)-6342, Olin Mathieson Chemical Corp.
ABSTRACT: Melam, melem, cyameluric acid, and melonic acid form a class of solid and infusible compounds known for their extreme heat stability. Experiments, to modify these structures by attaching alkyl, benzyl, phenyl and substituted phenyl groups aiming to produce still thermally stable, but liquid derivatives, are reported. Numerous substituted melams, melems, phenylcyamelurines, and esters of cyameluric acid have been prepared. Many preparations show excellent heat stability and have melting points in the range of 90 to 120°C; liquids have not been obtained. Derivatives of melonic acid could not be isolated.

WADD TR 60-357, OTS Release

September 1960

SUBJECT: MOLECULAR WEIGHT DETERMINATION IN THE CHARACTERIZATION OF POLYMERS
INVESTIGATOR: Richard M. Lange
ABSTRACT: Preliminary results are given on the determination of the macrostructure of poly-(dimethyl-2,2'-dimethylene pimelate) prepared by solution polymerization of 40% conversion. The physical methods used in this study include osmometry, light-scattering, viscometry and fractional precipitation.

Osmotic pressure measurements on a sample of the whole polymer in toluene solution yielded a number-average molecular weight of 3.36 x 10⁵ grams/mole. Light-scattering measurements on the same system gave a weight-average molecular weight of 2.80 x 10⁶ grams/mole. The heterogeneity index (M_w/M_n) of this polymer indicates a broad distribution of molecular weights.

WADC TR 53-373, Sup 8

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WADD TR 60-357, OTS Release (Continued)

A small sample of the polymer was subjected to fractional precipitation techniques with only partial success. Weight and number-average molecular weights were obtained on two fractions.

WADD TR 60-478, Part I

July 1960

SUBJECT: THE PREPARATION OF CERTAIN HETEROCYCLIC POLYMERS BY AN ALTERNATING INTRAMOLECULAR-INTERMOLECULAR CHAIN PROPAGATION
INVESTIGATOR: George B. Butler, K. Darrell Berlin, David L. Skinner, Robert W. Stackman
CONTRACT: AF 33(616)-5616, University of Florida
ABSTRACT: A number of additional monomers containing phosphorus and silicon have been synthesized and their polymerization studies. These monomers contain two non-conjugated double bonds in the 1,6-positions. All have been found to undergo polymerization by the intra-intermolecular chain propagation to produce soluble polymers. Some of the properties of the polymers are reported.

WADD TR 60-663, Part I, OTS Release

October 1960

SUBJECT: DETERMINATION OF RELATIVE STABILITY OF UREA COMPLEXES FROM X-RAY POWDER DIFFRACTION DATA
INVESTIGATOR: Jack Radell, J. W. Connolly
ABSTRACT: Urea complexes consist of a channel made up of hydrogen-bonded urea host molecules into which a variety of guest molecules can fit to form a crystalline complex. The stability of the resulting complex is a function of the size and properties of the guest molecule. A procedure has been devised which produces pure complex free of any uncomplexed urea or host molecules in solution. When the crystals of complex are removed from the solution, dissociation to urea and guest occurs to an extent dependent upon the dissociation constant of the complex. Characteristic interplanar spacings are obtained for a urea complex irrespective of the guest molecule present. The X-ray examination of a homologous series of compounds gives for each partially dissociated complex spacings for both urea and complex. The relative intensity of urea and complex spacings permits the estimation of the stability of such a complex compared to that of other homologues. The complexes of lower stability show a higher intensity ratio for tetragonal urea spacings/hexagonal complex spacings. It has been shown that the stability of a complex is greater for the higher members of a homologous series by this new application of X-ray data to several families of compounds. The X-ray data and stability was determined for several members of each of the following classes of compounds: alkyl-silanes, partially fluorinated esters, fumarates, maleates and acetylene derivatives.

WADD TR 60-700

July 1960

SUBJECT: CORRELATION BETWEEN STRUCTURE AND THERMAL STABILITY OF EPOXY RESINS
INVESTIGATOR: Gerhard F. L. Ehlers

WADC TR53-373, Sup 8

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WADD TR 60-700 (Continued)

ABSTRACT: The objective of this study was to survey the relationship between the structure and thermal stability of epoxy resins.

A basic epoxy resin from Bisphenol A, as well as a number of other di- and poly-epoxy resins of defined structure, were cured with equivalent amounts of various anhydrides, amines, phenols and catalysts. Weight loss of these resins was determined from periods up to 200 hours at 230°C, also the Vicat heat distortion temperature was determined before and after several aging periods.

Thermal stability, indicated by the weight loss data, and heat softening, indicated by the maximum heat distortion temperature obtained during the aging period, were correlated with the structures of the synthesized resins.

Rigid (aromatic) structures as well as high functionality of the reactants, or dense crosslinking were found to contribute to a high heat distortion. Anhydrides as curing agents were found to be more favorable in this respect than phenols and amines, because the reactivity towards epoxy as well as secondary hydroxyl groups resulted in higher crosslinking density.

Comparison of the three types of curing agents indicated about equal stability of the -C-O-C- and the -C-NH-C- linkage. Both were somewhat more stable than the ester linkage $\overset{\text{O}}{\text{C}}\text{-O-C-C}$.

Unexpected high heat softening points were obtained by using additives with one epoxy group and a double bond, such as dipenteneoxide, or curing agents, containing a double bond, such as maleic anhydride. The results obtained under certain conditions indicate that the double bonds apparently are polymerized due to the presence of epoxy groups, resulting in additional crosslinking.

WADD TR 60-870

February 1961

SUBJECT: INORGANIC HETEROPOLYMERS
INVESTIGATOR: Robert N. Hammer, Jack B. Kinsinger
CONTRACT: AF 33(616)-6943, Michigan State University
ABSTRACT: A kinetic study of the reaction of NaOSiO_3 with O_3SnCl in isobutanol has been undertaken. Sodium chloride forms rapidly and in approximately 100% yield, whereas $\text{O}_3\text{Si-O-SnO}_3$ is formed in 65-70% yield during the same period. Infrared evidence for the existence of secondary products exists. Earlier conductometric data for the reaction showed a gradual increase in resistance with time, but this now appears to result from slow aggregation of precipitated sodium chloride. A tentative mechanism for the reaction has been proposed.

WADD TR 60-870 (Continued)

The synthesis of siloxy-chromium compounds of the type $(R_3Si)_2CrO_4$ was undertaken preliminary to the study of polycondensation reactions. Methods developed for the preparation, isolation, purification, and characterization of these compounds were then used to study the more complex reactions of Cr(VI) compounds with silanediols. Both linear and cyclic siloxy-chromium compounds are believed to result from the reaction of silanediols with chromium (VI) oxide or chromyl chloride.

An attempt has been made to prepare siloxy-titanium compounds of the type $R_3SiOTi(R^1)_2OSiR_3$, where R = alkyl or aryl groups and R^1 = cyclopentadienyl groups. Competing side reactions, such as the formation of titanium dioxide or of $Ti(OSi\emptyset_3)_4$, apparently take place in preference to the intended condensation. Reports of similar studies elsewhere are conflicting, and therefore an investigation of the reaction mechanism is now under way.

The preparation of silylvanadates has been reviewed and the reported preparative techniques have been evaluated in this laboratory. Methods for the determination of chromium, titanium, vanadium, and silicon in transition metal-siloxyl compounds have been investigated.

ELASTOMERS

WADC TR 55-492, Part V

April 1960

SUBJECT: RESEARCH AND DEVELOPMENT ON HIGH TEMPERATURE FUEL RESISTANT RUBBER COMPOUNDS
INVESTIGATOR: Raymond G. Spain, Wayne G. Lajiness, Eva Deck
CONTRACT: AF 33(616)-5544, Wyandotte Chemicals Corporation
ABSTRACT: This report describes research directed toward the preparation of elastomeric vulcanizates resistant to high energy fuels (HEF) and hydro-carbon fuels.

Tests with HEF were made at temperatures up to 325°F, and tests involving the cycling of elastomeric vulcanizates in HEF and JP-6 utilized temperatures up to 450°F.

Tests of vulcanizates in JP-6 were made at temperatures up to 600°F using conventional testing procedures. Static and dynamic tests of experimental vulcanizates fabricated as O-rings were made at 400°F under simulated service conditions.

A variety of vulcanizing systems for fluoroelastomers was explored, and swelling studies of the vulcanizates indicated this technique to be applicable in the determination of the comparative degree of vulcanization of various specimens.

WADC TR 56-331, Part IV

April 1960

SUBJECT: RESEARCH ON HIGH TEMPERATURE RESISTANT RUBBER COMPOUNDS
INVESTIGATOR: Robert A. Hayes, Floyd M. Smith, Glenn A. Kidder, James C. Henning, Jack D. Rigby, George L. Hall
CONTRACT: AF 33(616)-3953, Firestone Tire & Rubber Company
ABSTRACT: Compounding of promising heat-stable elastomers to improve their physical properties has been continued. A representative series of 22 vulcanizates has been submitted to extensive high-temperature exposure conditions and tests which included: short term exposures, 3 or 4 minutes, in air at 600° and 800°F, in a nitrogen atmosphere with the sample in contact with a metal surface at 800°F and in nitrogen with the sample exposed to radiant heat from a metal surface 1/4" away at 1000°F; thermal conductivity measurements in the range of 100°F to 500°F; long-term aging, 300-500 hours at 300°F; and compression set determinations up to 500°F. Silicone, Viton, Kel-F 3700 and poly(ethyl acrylate) rubbers show outstanding thermal stability in many of these tests.

A method of constructing wirecord aircraft tires has been demonstrated and prototype tires of methacrylate and silicone rubber having excellent appearance were built. (Six additional methacrylate rubber tires were built under a separate WADC contract.)

WADC TR 56-331, Part IV (Continued)

The search for possible new high temperature elastomers has continued. A fairly heat-stable elastomeric polyester has been obtained from 5-pentoxyisophthalic acid and hexamethylene glycol and progress has been made in modifying this polymer to permit cures.

A new wholly aromatic polyamide fiber with outstanding high-temperature properties has been examined and methods for adhering it to various high-temperature vulcanizates were sought.

A bright aluminum coating applied by vacuum metallizing gave considerable surface protection to a resin-cured butyl vulcanizate exposed to 1000°F radiant heat in nitrogen.

WADC TR 57-651, Part III

May 1960

SUBJECT: RESEARCH ON RUEBERLIKE MATERIALS FOR APPLICATIONS INVOLVING CONTACT WITH LIQUID ROCKET PROPELLANTS
INVESTIGATOR: John H. Baldrige, Mark D. Inskeep
CONTRACT: AF 33(616)-5572, Connecticut Hard Rubber Company
ABSTRACT: Elastomeric materials resistant to hydrazine, unsymmetrical dimethyl hydrazine and JP-X fuel mixture have been found for service at room temperature and 160°F for immersion periods ranging from 21 days to 6 months. Among these is a new commercial polybutadiene elastomer. A semi-commercial elastomer resistant to n-propyl nitrate up to 3 months at 160°F has been found. Compounds which resist inhibited red fuming nitric acid up to 3 weeks at room temperature have been evaluated.

Several flexible plastics have been found resistant to nitrogen tetroxide (3 months), inhibiting red fuming nitric acid (3 weeks) at room temperature and liquid chlorine trifluoride at its boiling point (1 hour). No elastomers tested resisted nitrogen tetroxide or chlorine trifluoride.

Results are presented of fabrication studies, permeability tests and static immersion tests at 350°F and 400°F for resistant elastomers and plastics. A dynamic immersion test procedure has been developed, equipment assembled and a series of 24-hour evaluations run with several propellants.

WADC TR 59-338, Part II

April 1960

SUBJECT: COMPOSITE INORGANIC RESILIENT SEAL MATERIALS
INVESTIGATOR: M. Sabanas
CONTRACT: AF 33(616)-5793, Armour Research Foundation
ABSTRACT: The principal objective of this research program is to investigate and develop new concepts for novel and unconventional material combinations

WADC TR 59-338, Part II (Continued)

which would have resilience, recovery, strength, and chemical resistance at temperatures up to 1000°F. Major emphasis was given to configurations that would enable these materials to be used as static and dynamic reciprocating shaft seals.

Composite material combinations consisting of stainless steel and molybdenum fibers impregnated with tin, indium, magnesium, silver, and polymeric materials were produced.

Composites made of molybdenum fibers impregnated with silver were evaluated as static seals and showed good ability to seal air heated to 1000°F and retained pulsating pressures from zero to 5000 psi.

The relationship between the fiber structure, impregnant, and final composite were studied.

Impregnation techniques and secondary work processes like machining are outlined in this report.

WADC TR 59-749

April 1960

SUBJECT: COMPOSITE ELASTOMER-METAL O-RING SEALS
INVESTIGATOR: Jack R. Bos, Eugene B. Weber
CONTRACT: AF 33(616)-6182, Chance Vought Aircraft, Inc
ABSTRACT: A new approach to O-ring sealing has been attempted in order to improve the physical and mechanical properties of conventional elastomeric O-rings and eliminate the use of anti-extrusion devices. Elastomer-metal composite O-ring seals have been fabricated with a metallic spring concentrically embedded in the elastomer. Special molds and molding processes were required.

Performance evaluation of the prototype composite seals in hydraulic fluids at elevated temperature revealed that rolling or spiralling, a failure frequently experienced with conventional O-rings, was virtually eliminated. The spring cored O-ring performed much better in static impulse tests than in dynamic reciprocating tests. Seal failure in cycling tests resulted from excess nibbling and friction wear which were attributed to the greater force exerted on the sealing surface by the elastomer-metal O-ring. A limited investigation of decreased squeeze on the spring-cored O-ring seal showed improved cycling life.

WADD TN 60-154

September 1960

SUBJECT: HIGH TEMPERATURE FUEL TANK SEALANTS
INVESTIGATOR: Phillip A. House

WADC TR 53-373, Sup 8

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WADD TN 60-154 (Continued)

ABSTRACT: The Air Force has investigated the area of integral fuel tank sealants for advance design air weapons and has obtained materials which significantly advance the state-of-the-art. Fuel tank sealants are now available that will withstand considerable periods of aging in fuel and air at temperatures up to 600°F. A room temperature cure for fluoroelastomers has been discovered and efforts are now being directed toward suitable sealants using this curing system.

COMPOSITE MATERIALS

WADC TR 52-184, Sup 8

November 1960

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON
COMPOSITE CONSTRUCTION FOR FLIGHT VEHICLES
INVESTIGATOR: Donald G. Coleman
CONTRACT: AF 33(616)-58-1, Forest Products Laboratory
ABSTRACT: Developments in the program of research in composite construction for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1960 are summarized. In general, the approach has been to derive design criteria mathematically, and then to check by test. Three technical reports issued during the fiscal year are abstracted.

WADD TR 60-307, OTS Release

January 1960

SUBJECT: FORCED VIBRATIONS OF SANDWICH STRUCTURES
INVESTIGATOR: Alfred M. Freudenthal, Maciej P. Bieniek
CONTRACT: AF 33(616)-7042, Columbia University
ABSTRACT: In Part I of this report, a method is presented for the determination of the frequency response functions of the components of deformation and of stress in orthotropic sandwich plates. It applies to the case of simply supported rectangular plates loaded by dynamic pressure normal to their planes.

In Part II, a similar method is presented for orthotropic sandwich cylindrical shells. The boundaries of the shell are assumed as simply supported, and the dynamic pressure is normal to the middle surface. In both problems, the analysis takes into account the transverse shear deformation of the core and the material damping of core and facings. The results are presented in the form of expressions suitable for numerical evaluations.

ADHESIVES

WADD TR 59-15

April 1960

SUBJECT: DEVELOPMENT OF LOW PRESSURE AMBIENT TEMPERATURE CURING ADHESIVE FOR FIELD REPAIR OF METAL AIRFRAMES

INVESTIGATOR: Elizabeth A. Blommers, Earl W. Lane, Samuel Loshaek, Benjamin D. Halpern

CONTRACT: AF 33(616)-5984, Central Research Laboratory

ABSTRACT: Two-part room-temperature-curing adhesive systems, based on novolac epoxy resin EPIPHEN 825, were developed which pass the bend strength, the salt spray exposure, and all the shear strength requirements, except at -67°F , of Military Specification MIL-A-5090B when the adhesives are bonded to Alclad aluminum (cleaned in WAC-1 solution) and are cured for either six days at 73°F or 30 minutes at 212°F . The exceptional salt spray resistance of these adhesives was obtained by the incorporation of zinc or lead chromate as part of the filler. Heat-cures of one to two hours at 212°F of these systems can raise shear strengths at -67°F to 2500 psi, from 2000 psi, but salt spray resistance can thereby be lowered as much as 50%. A special cleaning procedure for aluminum, or the use of a four component adhesive, can eliminate the sensitivity to corrosion after a heat-cure.

One particular two-part high-temperature-resistant adhesive, when cured at temperatures between 200°F and 220°F can pass Military Specification MIL-A-5090B and also exhibit strengths of 1000 psi at 300°F and 400 psi at 500°F . Other high-temperature-resistant adhesives give shear strengths of around 1800 psi at 300°F but their other properties are not always up to the requirements of Military Specification MIL-A-5090B.

WADC TR 59-113, Part II

July 1960

SUBJECT: RESEARCH AND DEVELOPMENT ON INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND COMPOSITE CONSTRUCTIONS

INVESTIGATOR: Joseph Bayer, Ova E. Johnston, William A. Patterson

CONTRACT: AF 33(616)-5538, Aeronca Manufacturing Corporation

ABSTRACT: Modifications of the previously recommended inorganic adhesive were attempted to increase the bend resistance of the bonded joint. The modifications, incorporated singularly or in combination, include (1) substitution of various metal oxides and powders in the adhesive formula, (2) special surface preparation of the metal powders, (3) variation of the particle size and weight percentage of the powders, and (4) the introduction of porosity in the adhesive. Increased bend resistance was noted with the addition of phosphatized carbonyl iron and the addition of aluminum oxide. The aluminum oxide apparently introduced porosity into the adhesive. The processes which increased the bend resistance require further development to assure reproducibility of results.

WADC TR 59-113, Part II (Continued)

Methods for producing inorganic adhesive bonded honeycomb sandwich panels were further investigated. Preliminary experiments indicate that the quartz lamp-quench die method may alleviate some of the problems encountered in furnace bonding.

The development of new adhesives and processing techniques for bonding super alloys has been initiated.

WADD TR 60-687, OTS Release

November 1960

SUBJECT: AN INVESTIGATION OF LONGITUDINAL SHEAR DISTRIBUTION AND DAMPING IN A VISCOELASTIC ADHESIVE LAP JOINT
INVESTIGATOR: Carlos P. Avery
CONTRACT: AF 33(616)-6828, University of Minnesota
ABSTRACT: An analysis of the damping in a viscoelastic adhesive of a simple lap joint is presented. Expressions are developed for shear distribution and damping in the adhesive when the lap is sheared by a low frequency sinusoidal force. Calculations are made illustrating the variation in damping and shear distribution as a function of the dimensions and material properties of the lap components.

Experiments were performed which confirm the theory. Limitations, extensions, and possible refinements of the theory are presented.

EROSION

WADC TR 53-192, Part XV

June 1960

SUBJECT: MECHANISM OF RAIN EROSION - PART XV. Resistance of White Sapphire and Hot-Pressed Alumina to Collision with Liquid Drops

INVESTIGATOR: Olive G. Engel

CONTRACT: AF 33(616)-912, National Bureau of Standards

ABSTRACT: Data are presented that show the resistance of 0.318-cm (0.125-in) thick plates of white sapphire and hot-pressed alumina to impingement with 0.2-cm-diameter water-drops and mercury drops. In collision with mercury drops, the velocity at which damage was first observed was 3.514×10^4 cm/sec (1,153 ft/sec) for white sapphire and 4.276×10^4 cm/sec (1,403 ft/sec) for hot-pressed alumina; the difference in the velocities found for the two ceramics is not considered to be significant. The velocity required to damage these ceramic materials on collision with a waterdrop was not reached experimentally. A theoretical extrapolation suggests that plates of these ceramics of the indicated thickness can be expected to survive collision with 0.2-cm waterdrops without damage up to a velocity of 33.7×10^4 cm/sec (11,100 ft/sec). For air at 0°C, this is equivalent to a Mach Number of 10.

WADD TR 60-649

September 1960

SUBJECT: COMPARATIVE EROSION RESISTANCE OF PLASTIC MATERIALS IN A SUPERSONIC ROCKET EXHAUST AND SUBSONIC AIR ARC PLASMA

INVESTIGATOR: Herbert S. Schwartz

ABSTRACT: Representative thermally protective (ablative) plastics consisting primarily of organic and vitreous fiber reinforced materials were exposed in a hyperthermal subsonic air plasma stream and the supersonic exhaust of a rocket fueled with gaseous hydrogen and oxygen.

Linear and mass erosion rates were determined and correlated with materials composition. Relative performance of materials types in two different environments was obtained by establishing an erosion rate ratio parameter.

It was found that the class of materials having best performance was considerably different for the two exposure environments. In the rocket exhaust exposures, the best performing materials were high silica fiber, asbestos and glass fiber reinforced phenolics. In the subsonic arc plasma, the best performing materials were organic fiber reinforced phenolics.

PLASTICS

WADC TR 52-183, Sup 8

November 1960

SUBJECT: ANNUAL REPORT ON RESEARCH IN ANC-17 HANDBOOK PLASTICS FOR FLIGHT VEHICLES
INVESTIGATOR: Donald G. Coleman
CONTRACT: DO 33(616)-58-1, Forest Products Laboratory
ABSTRACT: Developments in the program of research in plastics for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1960 are summarized. In general, the approach has been to derive criteria mathematically, and then to check by test. One technical report was issued during the fiscal year and is abstracted.

WADC TR 53-192, Part XIV

May 1960

SUBJECT: MECHANISM OF RAIN EROSION - PART XIV. Pits in Metals Caused by Collision with Liquid Drops and Rigid Steel Spheres
INVESTIGATOR: Olive G. Engel
CONTRACT: AF 33(616)-59-3, National Bureau of Standards
ABSTRACT: A pit-depth-versus-velocity equation developed earlier for high-speed collision of liquid drops and soft, ductile metal spheres against targets of the soft and medium-hard metals was tested further with experimental data obtained using target plates of electrolytic tough pitch copper, 1100-0 aluminum, and 2024-0 aluminum, the static strength properties of which were measured by testing tensile specimens. The projectiles used to produce the pits were mercury drops, water drops, and steel spheres. It was found that the numerical constants in the equation are different for projectiles that flow during and as a result of the collision than for projectiles that do not flow (hardened steel spheres). Curves calculated by the equation were found to be in acceptable agreement with experimental pit-depth-versus-velocity data for collisions of the indicated projectiles with target plates of the three metals used, with the exception of the case of steel-sphere impingement against 2024-0 aluminum alloy. In this case work-hardening of the target metal seems to foster a mode of pit formation that was not considered in the development of the pit-depth-versus-velocity equation.

WADC TR 55-491, Part V

June 1960

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES
INVESTIGATOR: Gene H. Haertling, Kanaiyalal N. Parikh, H. Richard Thornton, Henry G. Lefort, Julian H. Lauchner, Dwight G. Bennett
CONTRACT: AF 33(616)-6192, University of Illinois

WADC TR 55-491, Part V (Continued)

ABSTRACT: Glassy bond and air setting adhesives were developed and studied. High temperature (to 2000°F) strength, ductility and moisture resistance were targets.

Methods of attack included metal and metal oxide powder additions, crystallization of gold nucleated bonding glasses, study of metal surface roughness function and evaluation of stress-strain relations in metal-adhesive composites.

In a study of moisture resistance, additions of about 33% of Fe_3O_4 were made to selected air setting or low temperature curing adhesives. Tensile shear values of the order of 1350 psi at room temperature and 1150 psi at 800°F were obtained but the adhesives disintegrated about a 3 1/2 hour boiling water treatment. When duplicate specimens were given a preliminary heat treatment of one hour at 1000°F shear strengths were reduced by about 50%, but they were essentially unchanged by the 3 1/2 hour boiling water treatment.

In glassy bond adhesives, stainless steel and phosphatized carbonyl iron powder additions gave improved strength, still increasing at 1000°F. Crystallized bonds gave higher strengths at the highest test temperatures. An optimum metal surface roughness value was noted. High order residual stresses (desirable to remove) were found in some metal-glassy bond composites.

WADC TN 59-251

July 1960

SUBJECT: DEGRADATION EFFECTS OF HIGH INTENSITY THERMAL RADIATION
ON PLASTIC RADOME MATERIALS

INVESTIGATOR: Rex W. Farmer

ABSTRACT: The thermal degradation characteristics of three representative reinforced plastic constructions were investigated at incident radiation intensities of 10 and 20 cal/cm²-sec using a carbon arc heat source.

The glass fabric reinforced materials studied included a polyester laminate and plastic sandwich construction with honeycomb and alkydisocyanate foam cores. Evaluation criteria, based upon visual examination of the test specimen, were defined in terms of the total incident energy required to cause various levels of degradation.

WADC TR 59-368, Part II

April 1960

SUBJECT: THE STUDY OF ABLATION OF STRUCTURAL PLASTIC MATERIALS

INVESTIGATOR: Franklin A. Vassallo, Norman E. Wahl, Gerald A. Sterbutzel,
John L. Beal

CONTRACT: AF 33(616)-5683, Cornell Aeronautical Laboratory, Inc

ABSTRACT: Results of ablation research conducted on reinforced plastic materials at moderately severe heating conditions are reported. The materials investigated include laminates of melamine, phenolic, and silicone resins reinforced with glass fabric as well as phenolic and silicone asbestos laminates. Experimental data are given for rate of material loss, effective heats of ablation, and depth of material degradation. The exposure apparatus provides a stream of high temperature nitrogen which is directed onto test specimens causing ablation to take place. The principal body shape considered is the 1/2" flat faced dowel.

High temperature exposures of composite bodies in which a substrate of high thermal conductivity is bonded to a layer of ablative material are reported. Experimental data include measurements of substrate temperature rise and rate of ablation. The temperature data are shown to be satisfied by the equation

$$\frac{T_s - T_0}{T_c - T_0} = 1 - \left[\left(1 - \frac{vt}{L_0}\right) \left\{ \frac{1 + \frac{5}{12}n}{1 + \frac{5}{12}n \left(1 - \frac{vt}{L_0}\right)} \right\} \right]^{\frac{n}{P_e}}$$

which is developed in the report. Discussion of heat sink effectiveness of the substrate is given.

WADC TR 59-459

June 1960

SUBJECT: EFFECTS OF HIGH TEMPERATURE, HIGH VELOCITY GASES ON PLASTIC MATERIALS
INVESTIGATOR: Frank P. Baltakis, Donald E. Hurd, Roy F. Holmes
CONTRACT: AF 33(616)-3421, Thermodynamics Laboratories
ABSTRACT: Twenty blunted, conical specimens of plastic materials were exposed to a Mach 1.82, 6300°F total temperature rocket exhaust jet to determine their erosion characteristics.

These specimens were composed of phenolic, epoxy, or Teflon resin in various proportions to Refrasil, extracted natural silicate, asbestos, glass, cotton, ceramic fiber, or nylon reinforcement in varied orientations.

The lowest stagnation point erosion rate was experienced by a Refrasil-phenolic specimen with "perpendicular-to-base" reinforcement orientation. The next lowest erosion rate was displayed by an extracted silicate-phenolic specimen with "cloth-wrap" reinforcement orientation.

Composition and properties of the erosion environment are presented.

SUBJECT: MECHANISM OF REINFORCEMENT OF FIBER-REINFORCED STRUCTURAL PLASTICS AND COMPOSITES

INVESTIGATOR: J. S. Islanger, K. Gutfreund, J. Brzuskiewicz, O. H. Olson

CONTRACT: AF 33(616)-5983, Armour Research Foundation

ABSTRACT: This is a continuation of the research effort undertaken to study the mechanism of reinforcement in fiber-reinforced structural plastics and composites. Analytical and experimental approaches include physical-chemical investigations of fiber-matrix interfaces, optical investigations of composites, studies of the mechanical aspects of reinforcement and investigations of the feasibility of graphite whiskers as reinforcements.

Data are presented for chemisorption studies aimed at determining the nature of interfacial bonds and the function of each component in the glass-surface treatment-resin composite. Macrosopic studies of fibers terminating within a resin are described, together with interference microscopy investigations.

Data are also presented for flexure tests of polyester specimens reinforced by a single layer of glass fibers located near one surface with fiber placement and treatment variations. A limited study of residual stresses in such a composite by analysis and by photoelastic methods is also presented.

The development of micro-tensile specimens for assessing the feasibility of graphite whiskers as reinforcements for plastics is described.

SUBJECT: DETERMINATION OF PROPERTIES OF SANDWICH MATERIALS SUBJECTED TO RAPID RISING TEMPERATURES SUMMARY TECHNICAL REPORT

CONTRACT: AF 33(616)-5762, Goodyear Aircraft Corporation

ABSTRACT: This report presents the results of an investigation of the mechanical properties of metal and plastic sandwich construction determined under rapid surface heating conditions for maximum surface temperatures up to 2000°F and maximum rate of surface temperature rise of 100°F to 200°F per second.

The evaluation of nine types of sandwich constructions included tests in the 650°F to 1000°F range for the plastic faced specimens, between 700°F and 900°F for aluminum and bonded stainless steel specimens, and 1300°F to 2000°F for the brazed and resistance welded types. The exposure periods at temperature ranged between 10 and 90 seconds. The testing loads selected ranged from 20 percent to 80 percent of room temperature ultimate strengths.

The comparative performance of the different groups of sandwich specimens can best be summarized by the results of the edgewise compression static tests. Deflection readings of the specimens were reported during the thermal shock period and are found in the tables.

WADC TR 59-668, Part II

February 1961

SUBJECT: A STUDY OF THE MECHANISM OF ABLATION OF REINFORCED PLASTICS
INVESTIGATOR: D. L. Chamberlain, Jr., D. E. VanSickle, C. W. Marynowski
CONTRACT: AF 33(616)-5964, Stanford Research Institute
ABSTRACT: Nonreinforced polyethylene, poly(methyl methacrylate) (Lucite), Teflon, and Phenolic resins were subjected to r-f discharges produced in nitrogen, oxygen, and air atmospheres. The gaseous products of degradation were determined, and possible mechanisms of attack are discussed.

Analyses of gaseous products resulting from arc-image furnace exposures on selected reinforced resins are reported.

A technique for sampling the gaseous boundary layer of an ablating resin in an argon-stabilized plasma jet is described and results are discussed. Boundary layer emission spectra, both line and band, are reported for plasma jet exposures of reinforced resins as a function of both viewing position and period of exposure.

WADD TR 60-24

March 1960

SUBJECT: HIGH MODULUS, HIGH TEMPERATURE GLASS FIBERS FOR REINFORCED PLASTICS
INVESTIGATOR: Philip J. Frickert, Ralph L. Tiede, Helmut I. Glaser, Allan B. Isham
CONTRACT: AF 33(616)-5802, Owens-Corning Fiberglas Corporation
ABSTRACT: A glass was developed which had a forth-five percent higher modulus of elasticity than the commercial glass used in the fibrous glass industry. Glass filament strands of this glass were produced at a rate of several pounds per hour from a single fiber-forming position and these strands were twisted and plied into yarns for weaving. 181-style cloth was woven, heat cleaned, and finished for the preparation of glass-plastic laminates. Strength and modulus of elasticity properties of laminates tested under standard conditions, and after fluid and thermal exposures, are reported.

WADD TR 60-72

May 1960

SUBJECT: IMPROVED HIGH TEMPERATURE RESISTANT TRANSPARENT PLASTIC MATERIALS
INVESTIGATOR: Earl E. Parker, Thomas L. St. Pierre, Walter E. Stickley, Edward A. George, Michael R. Dzugan
CONTRACT: AF 33(616)-5602, Pittsburgh Plate Glass Company
ABSTRACT: This research effort was initiated in an effort to improve the high temperature capabilities of one chemical type of optically transparent plastic, for use in Air Force flight vehicle glazings. Specific improved formulations achieved many of the mechanical and optical target properties.

WADD TR 60-72 (Continued)

Attempts to orient the improved formulations toward a resistance to crack propagation met with very limited success. Detailed descriptions of the orientation procedures developed and subsequent results are included.

WADD TR 60-101

September 1960

SUBJECT: CONFERENCE ON BEHAVIOR OF PLASTICS IN ADVANCED FLIGHT VEHICLE ENVIRONMENTS
INVESTIGATOR: H. S. Schwartz
ABSTRACT: This report is the collection of papers presented at the Materials Central, WADD conference on "Behavior of Plastics in Advanced Flight Vehicle Environments" held in Dayton, Ohio, on 16-17 February 1960.

The purpose of this conference was to review the recent progress in studies on plastics exposed to severe thermal environments and simulated extremely high altitude environments. The papers presented are primarily on internal and contractual programs sponsored by the Plastics Branch of the Nonmetallic Materials Laboratory. Papers on work performed by other organizations in the Department of Defense and by their contractors were also presented and are included.

WADD TR 60-110

July 1960

SUBJECT: THERMAL PROTECTIVE SURFACES FOR STRUCTURAL PLASTICS
INVESTIGATOR: James W. Vogan
CONTRACT: AF 33(616)-6393, Bendix Products Division
ABSTRACT: An exploratory materials research program has been conducted on high temperature insulating and erosion resistant surface materials, which are suitable for short time thermal protection of structural plastics.

New heat insulating material composite systems were investigated. These multicomponent structural elements were composed of an outer refractory surface layer, a thin metallic film, a porous inorganic heat insulating layer and various substrate plastics, with all elements integrally, mechanically bonded together.

The individual components of the protective surfaces were evaluated for desirable properties using subsonic and supersonic high temperature gas streams. With the aid of these results composite heat-protective surfaces were formulated and evaluated in the same test environments. Experimental results showed a significant lag in heat penetration into the substrate plastic during hyperthermal exposure. Tungsten faced ceramics exhibited good thermal erosion resistance and heat insulation in high temperature reducing atmospheres, and nickel-zirconia protective surfaces were optimum in high temperature oxidizing environments.

WADD TR 60-110 (Continued)

The principal problems encountered with the heat insulating composite systems were suitable methods of fabrication and maintenance of structural integrity in the interface regions during test exposure. Considerable progress was achieved on these two problem areas, and technical effort is continuing. In addition, new and improved heat protective surfaces and exploratory methods of fabrication were investigated.

WADD TR 60-125

July 1960

SUBJECT: THE EFFECTS OF HIGH VACUUM AND ULTRAVIOLET RADIATION ON PLASTIC MATERIALS
INVESTIGATOR: Norman E. Wahl, Roy R. Lapp
CONTRACT: AF 33(616)-6267, Cornell Aeronautical Laboratory, Inc
ABSTRACT: This study was concerned with the behavior of plastic materials exposed to simulated conditions of pressure, temperature and near ultraviolet as encountered outside the earth's atmosphere.

Glass fiber reinforced plastic laminates, of three types, were exposed to vacua in the order of 10^{-6} to 10^{-7} mm Hg and ultraviolet flux ranging from 1/3 to 2-1/2 times the solar constant of 2 calories $\text{cm}^{-2} \text{ min}^{-1}$. The periods of exposure ranged from 3 to 500 hours. The equilibrium temperatures of the laminates varied from 250° to 465°F depending on the intensity of the ultraviolet employed.

After exposure to ultraviolet radiation equivalent to the solar constant and vacuum, for periods up to 500 hours, no great change in dimensions or loss in weight of the laminates was observed. The strength of the polyester specimens, however, tends to increase while the phenolic and epoxy laminates decrease slightly in strength.

Laminates exposed to ultraviolet flux of 4.9 calories $\text{cm}^{-2} \text{ min}^{-1}$ and vacuum for three hours show considerable loss in weight and flexural strength.

WADD TR 60-176

October 1960

SUBJECT: FRACTURE STUDIES OF TRANSPARENT AIRCRAFT MATERIALS
INVESTIGATOR: Herschel L. Smith, William E. Anderson
CONTRACT: AF 33(616)-57-32 and AF 33(616)-58-16
ABSTRACT: This study was initiated to assess the relative shatter resistance of stretch oriented, as-cast, and laminated acrylics as a function of temperature and material thickness.

Simulated air frame enclosure conditions were used in the laboratory program. Formed semi-cylinders, internally pressurized, and under controlled temperatures were subjected to impact and penetration by high velocity projectiles.

WADD TR 60-176 (Continued)

The stretch oriented acrylic exhibited superior impact fracture resistance over the entire temperature range investigated. In addition, the results indicate that substantial weight savings are possible (35% to greater than 65%) without loss of structural integrity or reduced shatter resistance by using stretched acrylics in lieu of as-cast or laminated materials. Individual results are presented.

WADD TR 60-177, Part I

June 1960

SUBJECT: STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES - PART I. Phenolic-Asbestos, R/M Pyrotex Felt Style 41-RPD

INVESTIGATOR: Kenneth H. Boller

CONTRACT: DO 33(616)-58-1, Forest Products Laboratory

ABSTRACT: Several reinforced plastic laminates that show promise of having good strength properties at elevated temperatures are being tested to determine their strength and elastic properties. This report, the third of the series, presents results of mechanical tests on a phenolic resin laminate reinforced with an asbestos felt (R/M Pyrotex Felt Style 41-RPD). Data were obtained after exposure periods of up to 1,000 hours from such tests as weight loss, flexure, tension, compression, interlaminar shear, and bearing. Most of the tests were made parallel to the machine direction of the asbestos sheets to determine the effects of high temperature and time on the strength properties. Tension tests were made at both 0° and 45° to machine direction to obtain data from which edgewise shear strength and modulus of rigidity can be calculated. Creep and stress-rupture data were obtained under both tension and compression loads.

In general, the strength properties of the phenolic-asbestos laminate decrease with increases in temperature. Specifically, the strength properties decrease with the first application of heat but then often increase with continued exposure or increased temperatures up to 500°F . Finally, after some critical time at temperature, the properties decrease with additional time at temperature. All tests except interlaminar shear show that over 50 percent of the room-temperature strength was retained when laminates were exposed for 1/2 hour at temperatures up to 800°F . The specific magnitude of these strengths are presented in tables and curves.

WADD TR 60-202

September 1960

SUBJECT: EFFECT OF ENVIRONMENT ON MECHANICAL PROPERTIES OF GLASS FABRIC PLASTIC SANDWICH

INVESTIGATOR: P. M. Jenkinson, E. W. Keunzi

CONTRACT: DO 33(616)-58-1, Forest Products Laboratory

WADD TR 60-202 (Continued)

ABSTRACT: This report presents the results of flatwise flexure and edgewise compression tests on three constructions of glass-fabric plastic sandwich that were exposed at the seashore in south Florida for 3 months and for 1 year. Included are results of tests of sandwich panels that were subjected in the laboratory to 100°F and 100 percent relative humidity for 16 months. The three constructions had facings approximately 0.03 inch thick of glass-fabric laminate treated with polyester, epoxy, and heat-resistant phenolic resins. Two panels of polyester sandwich were evaluated, one painted and one unpainted. Cores were of glass-fabric honey-comb treated with the same type of resin as the facings except that the core for the sandwich with epoxy resin facings was treated with nylon-phenolic resin.

Tests of all constructions were made at room temperatures, and also at 500°F for the heat resistant sandwich.

The unpainted polyester sandwich panel deteriorated the most in 1 year's weathering, with both edgewise compressive and shear strengths being reduced 14 percent. Painting the other polyester panel with a MIL-E-7729 enamel of the type normally used on exterior reinforced plastic aircraft parts prevented loss in edgewise compressive strength and reduced the loss in shear strength to 6 percent. In comparison, unpainted polyester solid laminate panel 1/8-inch thick also had compressive strength reduced 14 percent by 1 year's weathering.

At 500°F the heat-resistant phenolic sandwich panel showed a 13 percent increase in edgewise compressive strength after a year's weathering, compared to the control value at 500°F.

Exposure to 100°F, 100 percent relative humidity for 16 months reduced edgewise compressive strength by 47 percent for the unpainted polyester sandwich panel, 51 percent for the painted polyester sandwich panel, 24 percent for the epoxy sandwich panel, and 29 percent for the heat-resistant phenolic sandwich panel. Exposure of 1/8-inch thick solid laminates to 100°F and 100 percent relative humidity for 1 month reduced compressive strengths 27 percent for polyester, 13 percent for epoxy, and 15 percent for phenolic laminates.

WADD TR 60-203

April 1960

SUBJECT: OPTICALLY TRANSPARENT MATERIALS FOR SPACE FLIGHT VEHICLES
INVESTIGATOR: Robert E. Wittman
ABSTRACT: Requirements for optically transparent materials in manned re-entry, orbital or general space flight vehicles for human vision are outlined.

A general survey to define the conditions imposed by natural and induced environments on these materials is reported.

WADD TR 60-203 (Continued)

The present capabilities of transparent materials as related to the predicted environments to be imposed on space flight vehicles is included. Resultant conclusions for research and development to improve certain properties of presently available optically transparent materials are given.

WADD TN 60-287

November 1960

SUBJECT: PROPERTIES OF HIGH MODULUS REINFORCED PLASTICS
INVESTIGATOR: George P. Peterson
ABSTRACT Properties of laminates made with a new high modulus glass fiber designated as YM31A glass, developed by the Owens-Corning Fiberglas Corporation under Materials Central contract, were determined. The work consisted of a determination of the mechanical and electrical properties under a variety of environmental conditions utilizing an optimum resin-finish combination.

The investigation demonstrated that YM31A laminates not only increased the modulus from 25% to 45% over duplicate "E" glass laminates but also had equivalent or higher strength properties. The electrical properties in the lower temperature ranges were approximately equivalent but showed a greater change in dielectric constant with increased temperature in the 200°-500°C range.

Factors which greatly increase the effectiveness of YM31A from both a strength and modulus aspect are minimizing yarn constraint (less interlacing in fabric pattern) and the use of resin systems, such as the epoxy type, which are not only more ductile but exhibit a high adhesive bond strength between resin and glass.

WADD TR 60-314

September 1960

SUBJECT: THE THERMAL DIFFUSIVITY OF A STRUCTURAL PLASTIC LAMINATE AT ELEVATED TEMPERATURES
INVESTIGATOR: Rex W. Farmer
ABSTRACT: The applicability of previously developed relations for dynamically measuring the thermal diffusivity of structural plastic laminates is briefly examined. The thermal diffusivity was found to be an "apparent" or "effective" value for these materials above their degradation temperature with accompanying heat of reaction effects. Some exploratory experiments at temperatures up to 1,000°C are described for a phenolic resin/glass fabric laminate using a radiant heating source.

WADD TR 60-380

June 1960

SUBJECT: REINFORCED PLASTICS AT VERY HIGH TEMPERATURES

WADD TR 60-380 (Continued)

INVESTIGATOR: Donald L. Schmidt, George P. Peterson

ABSTRACT: The recent development of thermally stable reinforced plastics is an important contribution to the advancement of high temperature materials technology. Heat resistant reinforced plastics are presently available for extended use at temperatures up to about 500°F, and short-time use at temperatures up to about 1,000°F. These materials also have structural-insulative capabilities in extremely high temperature environments (over 5,500°F), like those encountered in hypersonic re-entry and advanced propulsion systems.

A general review of high temperature reinforced plastic materials is presented in this report. Structural properties and characteristics of various plastics at temperatures up to 1000°F are discussed, using illustrative data. The behavior of reinforced plastics in very high temperature environments is briefly reviewed. Performance characteristics are given in terms of both materials and environmental variables.

WADD TR 60-393, OTS Release

September 1960

SUBJECT: EVALUATION OF BRAZED HONEYCOMB STRUCTURES

INVESTIGATOR: Robert C. McMaster, Anthony T. D'Annessa, Henry W. Babel

CONTRACT: AF 33(616)-6364, The Ohio State University

ABSTRACT: The final report on U. S. Air Force Contract No. AF 33(616)-6364, administered by the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, summarizes the results of a one-year study of nondestructive test methods for the examination of brazed and welded joints in complex shapes. The investigation was directed, by mutual agreement, to commercially-available nondestructive test methods for evaluation of the quality and serviceability of brazed honeycomb structures. Applicable nondestructive test methods were found to include (1) penetrating radiation tests, (2) ultrasonic tests, and (3) thermal tests. Numerous experimental and production parts and brazed honeycomb assemblies provided by government and industrial facilities were examined by each test method and the results were correlated and evaluated. It was found that several commercially-available nondestructive test methods provide reliable and detailed indications of surface layer discontinuities such as core-to-skin filleting and metal-to-metal bonds in edge members. X-ray test methods alone provided reliable and detailed indications of internal conditions such as crushed core, node flow, vertical-tie braze areas, and repaired cells.

Penetrating radiation test methods evaluated included: (1) X-ray film radiography, (2) xeroradiography, (3) direct fluoroscopy, (4) fluoroscopy with television image amplifiers, (5) fluoroscopy with electrostatic image amplifiers, (6) large-area photoconductive screen television image amplifiers, and (7) small-area photoconductive screen television image enlargers. Under properly-applied and controlled test conditions, each of these X-ray inspection systems was found useable in the inspection of brazed honeycomb structures.

Ultrasonic test methods evaluated included: (1) immersion ultrasonic tests with focussed transducers, (2) contact ultrasonic tests with resonance transducers, and (3) contact ultrasonic tests with pulse-reflection transducer systems. Detailed images of surface-layer conditions and discontinuities were obtained only with immersion ultrasonic tests with focussed transducers and high-resolution C-scan facsimile or memory-tube recorders. Large-area voids were detectable, under carefully-controlled test conditions, with contact ultrasonic tests with swept-frequency resonance transducer systems.

Thermal test methods evaluated included: (1) thermal-flash proof tests, (2) thermal-gradient tests with heat-repelled fluid coatings, (3) thermal-gradient tests with thermographic phosphor coatings, (4) thermal tests with temperature-sensitive color-changing paint coatings, and (5) thermal tests with remote infrared sensing systems. Reliable detailed images of surface-layer conditions and discontinuities were obtained with thermal gradient tests with transient front-side panel heating and heat-repelled fluid coatings that produced permanent images on the surfaces of the test specimens. Large-area voids were detected by bulging of the skin materials in thermal-flash proof tests. Other thermal test methods described are experimental in nature and deserve consideration for further development.

Complete detail indications have been confirmed for numerous test specimens whose surface-layer discontinuities were revealed by proper application and control of the following nondestructive test methods:

1. Film radiography
2. Xeroradiography
3. Direct fluoroscopy
4. Fluoroscopy with electrostatic image tubes
5. Fluoroscopy with photoconductive-screen television systems
6. Immersion ultrasonic testing with focussed transducers and C-scan facsimile recording systems
7. Thermal-gradient tests with front-side transient heating of panels coated with heat-repelled fluid media

Numerous specimens containing surface-layer discontinuities have been subjected to destructive peel tests or to metallographic sectioning and examination. The results of these destructive examinations confirm the nondestructive test indications produced by the methods listed above.

Further research and development are recommended for the following experimental nondestructive test methods:

1. Xeroradiography
2. Photoconductive-screen television systems, particularly those which permit image enlargement without deleterious loss of image detail
3. Thermal-gradient nondestructive tests with both direct coatings or remote sensing systems which respond to thermal gradients rather than to absolute temperatures.

WADD TR 60-393 (Continued)

Although brazed honeycomb assemblies can be inspected reliably and in detail by methods commercially available at present, the most effective test systems are presently slow and costly. Further developments are recommended for those systems which offer promise of adequate detail sensitivity and reliability, but with which the tests can be made faster and at lower costs.

WADD TR 60-402

August 1960

SUBJECT: EVALUATION OF BIAXIALLY STRETCHED TRANSPARENT PLASTICS
INVESTIGATOR: D. A. Hurst
CONTRACT: AF 33(616)-3994, Rohm & Haas Company
ABSTRACT: This program was conducted in an effort to improve the crack propagation resistance by hot stretching methods, of a developmental thermosetting type of transparent plastic material. This material, a modified acrylic known as Selectron 400, has an elevated temperature structural capability well above that of any plastic glazing material available in quantity today.

Conditions under which Selectron 400 can be stretched without breaking were very critical and were not fully successful. Three basic processing steps were required; hot stretching without causing an appreciable amount of cross-linking; relatively low temperature irradiation to cause sufficient cross-linking of the stretched material to prevent relaxation of the degree of stretch at elevated temperatures; oven treatment at an elevated temperature to produce the cross-linking action. A great deal of tearing and breakage of sheets resulted in very low yields of stretched material. The effect of varying the stretching conditions were studied with only mild success in improvement.

Crack propagation resistance of the cross-linked stretched Selectron 400 was very poor. In order to determine whether the limited amount of stretching possible to obtain had any significant effect on the crack propagation resistance, tests were conducted on stretched material without cross-linking. The results indicated that hot stretching essentially produced no gain in crack propagation resistance.

WADD TR 60-466

September 1960

SUBJECT: STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES (Epon 1031 Resin and 181-Volan A Glass Fabric)
INVESTIGATOR: Kenneth H. Boller
CONTRACT: DO 33(616)-58-1, Forest Products Laboratory
ABSTRACT: Several reinforced plastic laminates that show promise of having good strength properties at elevated temperatures are being tested to determine their strength and elastic properties. This report, the fourth of the series, presents results of mechanical tests on an epoxy resin laminate

WADD TR 60-466 (Continued)

reinforced with glass fabric (Epon 1031 with 181-Volan A). Data were obtained after exposure periods from 2 minutes to 1,000 hours from such tests as weight loss, flexure, tension, compression, interlaminar shear, and bearing. Most of the test were made parallel to the warp of the glass fabric to determine the effects of time and above-normal temperatures on the strength properties. Tension tests, however, were made at both 0° and 45° to the warp to obtain data from which edgewise shear strength and modulus of rigidity can be calculated. Creep and stress-rupture data were obtained under both tension and compression loads.

In general, the strength properties of this epoxy glass fabric laminate decrease with increase in temperature at constant periods of exposure. Specifically, however, the resin softens and the strength of the laminate decreases with the first application of heat, but then at constant temperature the strength properties often increase. Finally, after some critical time, additional exposure causes the properties to decrease. The specific magnitude of these strengths are presented in tables and curves.

WADD TR 60-648

November 1960

SUBJECT: ABLATIVE BEHAVIOR OF PLASTICS IN SUBSONIC AND SUPERSONIC HYPERTHERMAL FLOW

INVESTIGATOR: Rex W. Farmer

ABSTRACT: Materials research was conducted on the ablative behavior of four plastic materials in subsonic and supersonic hyperthermal air/argon environments. Small hemisphere cylindrical models of the experimental materials were exposed to the very high temperature gas stream, which was generated by a stabilized electric arc. Environmental conditions used were: stagnation enthalpies from 750 to 2,600 Btu/lb, free stream Mach numbers of 0.9 and 1.8, and stagnation pressures of 1.8 and 4.0 atmospheres.

The material behavior was found to vary with certain environmental and materials parameters. Within the range of environmental conditions studied, inorganic fiber reinforced phenolic resin materials had the lowest erosion rates in supersonic flow while the lowest erosion rates in subsonic low mass flow were observed for a phenolic/nylon material.

FIBROUS MATERIALS

WADD TR 60-9, OTS Release

June 1960

SUBJECT: A POSSIBLE APPLICATION OF ORGANIC FIBERS IN HIGH TEMPERATURE ENVIRONMENT

INVESTIGATOR: Myron J. Coplan, Donald H. Powers, Jr., Leo Barish, Emery I. Valko

CONTRACT: AF 33(616)-6234, Fabric Research Laboratories, Inc.

ABSTRACT: It is taken as a premise that fabrics used in re-entry drag parachutes (1) require typical textile properties up to deployment but (2) that they need not survive in their original chemical state subsequent to deployment provided the decay occasioned by re-entry conditions (heat, active oxygen, irradiation) does not interfere with the deceleration function. The possible utility of linear organic polymers as the principal chemical species in such materials is examined.

Details of re-entry environment conditions, as applicable to the problem, are developed. Some of the textile qualities required of the parachute fabric up to deployment are reduced to quantitative criteria. Typical organic, metallic and ceramic materials are contrasted and the advantages of the first class are defined.

Recognizing that the principal deficiency of the organic polymer fibers resides in their thermal and thermochemical sensitivity, a survey of the causes of these deficiencies is given. Some speculative arguments are advanced relative to means of overcoming the deficiencies. In addition, a broad concept for a means of employing the organic materials is proposed wherein thermal degeneration is to be anticipated and even used to advantage.

A detailed account of the principles of the so-called "metamorphic" fiber system is given. A number of specific thermochemical reaction schemes and polymer systems are proposed.

Unqualified conclusions regarding the feasibility of the basic principle and any of the specific schemes are not possible in the light of the documented need for further experimental effort.

WADD TR 60-150, OTS Release

May 1960

SUBJECT: DEVELOPMENT OF SHADE STANDARDS AND TOLERANCES FOR USAF BLUE 84 AND BLUE 584 SERVICE AND OPTIONAL WEAR UNIFORMS

INVESTIGATOR: Frank J. Rizzo, Constantine J. Negas, Alvin O. Remsley, William B. Bushnell, Ruth J. Evans

CONTRACT: AF 33(616)-53-221 and 54-2, Quartermaster Research and Engineering Command, U. S. Army

ABSTRACT: The report covers the development of a number of shade standards and tolerances in USAF Blue 84 used by the USAF in the service uniform and also certain optional wear uniforms, and for Blue 584 used in special components. The standardization procedure developed by the Quartermaster Corps along concepts which have been common in the industry for many years was applied to this development.

WADD TR 60-150, OTS Release (Continued)

As part of this work, an extensive study of dye formulations was accomplished from which new highly colorfast formulations were developed. These formulations, in addition to fulfilling the basic colorfastness requirements, have application characteristics suitable for both large and small industry.

The formulation selected represents somewhat of a compromise, a situation which is normal in the usual interaction between colorfastness properties, application characteristics, availability factors, and end use requirements.

The complete program was monitored by application of both standard visual techniques and by colorimetric analysis based on spectrophotometric data transposed in accordance with the tristimulus functions of the CIE standard observer for colorimetry.

The tolerance ranges which have been developed around each of the standards in the several different fabric constructions are visually well spaced, although the colorimetric spacing of the instrumental technique does not coincide with visual judgment. The reasons for this situation are analyzed, and it is concluded that a further review of the theory of color mixtures must be made for the type of blending operations which are involved in a program of shade standard and tolerances of the type being reported.

WADD TR 60-151, OTS Release

June 1960

SUBJECT: DEVELOPMENT OF SHADE STANDARDS AND TOLERANCES FOR SILVER TAN 193

INVESTIGATOR: Frank J. Rizzo, Constantine J. Megas, Alvin O. Ramsley, John T. Walwood, Ruth J. Evans

CONTRACT: AF 33(616)-53-221 and 54-107, Quartermaster Research and Engineering Command, U. S. Army

ABSTRACT: Shade standards and tolerances have been developed for tropical and gabardine weave fabrics in all wool, wool/polyester and polyester/rayon blends. Tolerance spacing is such as to permit interchange of uniform components made for the blend fabrics with components made of all wool so long as the weave construction is common. The limiting factor to such usage is considered to be differences in appearance originating with the gloss of the fibers and not the chromatic and lighness deviations of the tolerance range. Colorfastness is good at 40 Standard Fading Hours for all fabrics, except for the polyester/rayon tropical where the vat dyed rayon permits a rating of good at 120 Standard Fading Hours. Dye formulations selected are practical for routing application and stable to finishing factors.

WADD TR 60-198, OTS Release

June 1960

SUBJECT: DEVELOPMENT OF A FABRIC TO PROVIDE PERSONAL PROTECTION FROM TOXIC MATERIALS

INVESTIGATOR: Raymond G. Spain, Alexander V. Sanger

WALC TR 53-373, Sup 8

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WADD TR 60-198 (Continued)

CONTRACT: AF 33(616)-6501, Wyandotte Chemical Corporation

ABSTRACT: This report describes research directed to the development of a coated fabric to resist the chemical effects of and be impermeable to high energy fuels (HEF).

This program included the design and fabrication of special test equipment and of a laboratory fabric coating apparatus.

A base fabric (Dacron) was selected on the basis of resistance to HEF, and the majority of the effort was directed toward the coating of this fabric with fluoroelastomer based compositions and the necessary subsequent testing.

A coated fabric was approved and 100 yds.² of this material were prepared and delivered to the Nonmetallic Materials Laboratory, Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division.

WADD TR 60-244, OTS Release

June 1960

SUBJECT: CONTINUOUS FILAMENT CERAMIC FIBERS

INVESTIGATOR: W. A. Lembertson, D. B. Aiken, E. H. Girard

CONTRACT: AF 33(616)-6246, Carborundum Company

ABSTRACT: Melting and fining of refractory glass compositions and drawing of continuous filament ceramic fibers have been carried out in a dry, inert-atmosphere glovebox, which permitted the use of refractory metals, graphite, and boron nitride as crucible and heater materials, and prevented the adsorption of moisture by the filaments.

Several refractory glasses in the baria-alumina-silica, calcia-alumina-silica, and magnesia-alumina-silica systems were successfully drawn into continuous filaments. Tensile strengths of filaments drawn were determined at room and elevated temperatures. Values as high as 138,000 pounds per square inch at room temperature and 132,000 pounds per square inch at 1500°F (815°C) were obtained.

WADD TR 60-252, OTS Release

October 1960

SUBJECT: THE DESIGN AND EVALUATION OF HEAT STABILIZED TAPES AND WEBS

INVESTIGATOR: Jacob Serbin, Herman Becker

CONTRACT: AF 33(616)-39447, Narricot Corporation

ABSTRACT: The basic purpose of this work was to design and evaluate a series of 13 representative webbings, tapes and ribbons, which would maintain maximum breaking strength and minimum shrinkage when subjected to a temperature of 250°F for a period of five hours.

Each of the 13 samples were to be made from:

1. Dupont Type 330 or 700 nylon

WADD TR 60-252 (Continued)

2. Chemstrand Type RMB nylon
3. Dupont Type 51 or 52 Dacron

A heat stabilization process was found necessary to accomplish the desired shrinkage. An experimental pilot program, involving three types of stabilization processes, indicated that a "heat stabilized" continuous method (HSCM) using a dry air oven was the most practical method.

Investigation has shown that, with due consideration given to the strength per weight ratio, a woven end item of nylon or Dacron can be stabilized with a measureable degree of success. Additional information was sought to ascertain strength and shrinkage of all items at room temperatures, and also after five hours at 300°F. Dacron items only were tested after one hour exposure at 350°F. All items were checked for seam efficiencies in the natural state and after exposure to 250°F and 300°F for five hours.

Fifty (50) yards of each of the 39 samples were delivered to WADD for evaluation. The decision was made to use nylon materials. The complete set of thirteen (13) samples were finally selected from among the Dupont or Chemstrand nylon webbings, tapes and ribbons.

WADD TN 60-253, OTS Release

December 1960

SUBJECT: ULTRA VIOLET RADIATION RESISTANT MATERIALS FOR DECELERATION
INVESTIGATOR: Joyce C. McGrath
ABSTRACT: Two improved types of nylon yarns (1) Type 330, 30 denier, a more ultra-violet radiation and heat resistant yarn and (2) Type 300, 30 denier, a higher strength than the conventional nylon yarn, were developed by the E. I. duPont deNemours and Company, Incorporated, to be used in materials for personnel and other type decelerators.

Cloth, woven of the two types of nylon yarn, in both natural and International Orange were exposed to (1) outdoor weathering at the Naval Auxiliary Air Station, El Centro, California for periods of 1, 2, 3, 4, 5 and 6 weeks, (2) Accelerated aging in a Weather-O-Meter, Type XIA, for periods of 20, 40, 60, 80, 100 and 150 hours and (3) a temperature of 300°F for 8 to 16 hours.

Data obtained indicates that the Type 330 nylon cloth in natural color retains a very high percentage of strength under all three exposure conditions while the Type 330, International Orange and the Type 300 in both natural and International Orange deteriorated at a very rapid rate after exposure to both outdoor weathering and accelerated aging. The Type 300, natural showed the greatest loss in strength after heat exposure of 8 to 16 hours.

(It was determined, in supplemental tests, by the E. I. duPont deNemours Company that the type of dye used to obtain the International Orange color, was the chief factor in strength loss of the dyed Type 330 cloth. The type 300 was not developed as a heat or ultra-violet light resistant yarn).

SUBJECT: DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR USAF TAN 505

INVESTIGATOR: Frank J. Rizzo, Constantine J. Megas, Alvin O. Ramsley, William B. Bushnell, Ruth J. Evans

CONTRACT: AF 33(616)-53-221; 55-72, Quartermaster Research and Engineering Command

ABSTRACT: Shade standards and tolerances for USAF Tan 505 used in hot weather clothing have been developed in three fabric constructions using a formulation which has had considerable industrial application for a number of related shades.

The shade standardization procedure used was based on a technique developed at the Quartermaster Research & Engineering Command, following procedures which are well known in the Industry.

The program was monitored by visual as well as colorimetric procedures. The tolerance spacing visually should provide for a reasonable approach to a monotone appearance in uniforms made from two different pieces of fabric in the same construction.

The shade characteristics of the three fabrics are such as to provide for use of any of the fabrics in a single ensemble. Whatever differences occur are more related to fabric texture than to specific differences in color. The colorimetric spacing obtained instrumentally does not coincide with the visual evaluation. The report discusses the reasons for this in detail.

The degree of colorfastness achieved is optimum for the depth of shade.

SUBJECT: DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR USAF BLUE 157 NYLON RAYON POPLIN AND POLYVINYL BUTYRAL COATED NYLON TWILL

INVESTIGATOR: Frank J. Rizzo, Constantine J. Megas, Alvin O. Ramsley, Ruth J. Evans

CONTRACT: AF 33(616)-53-221 & MIPR (33-616)56-15, Quartermaster Research & Engineering Command

ABSTRACT: Shade standards and tolerances for Blue 157 have been developed in two separate fabric constructions used in USAF rainwear, namely, the cloth, nylon-rayon poplin and the cloth, rayon, twill, PVB coated. The formulation used for the former fabric was one prescribed by the USAF. The formulation of the latter was one which was selected after a rather extensive study of colorants. The durability of the formulation of the nylon-rayon poplin to degradative factors is dependent upon the effectiveness of the durable water repellent treatment. The formulation used on the nylon twill has inherently good colorfastness. The data reported show that good colorfastness was obtained in both instances. The tolerance spacing and general standardization procedure utilized was one developed by the Quartermaster Research and Engineering Command following procedures which are well known in the industry.

WADD TR 60-295, OTS Release (Continued)

The program was monitored by both instrumental and visual methods and the tolerance spacing provided is such as to be in harmony with the essential military requirements of good appearance and the recognized capabilities of the industry. The tolerance spacing on the nylon-rayon fabric is slightly wider than that on the coated fabric. This was occasioned by the greater complexity of the dyeing system required for an ortho blend against a single fiber system. In addition, coating tends to a narrowing of the shade range from the precoat condition. These tolerances as spaced colorimetrically are different from their visual appearance. The latter has been accepted as the important consideration in light of the fact that the colorimetric theory is under question. Some of the possible reasons for these deviations are enumerated in the report.

WADD TR 60-385, OTS Release

July 1960

SUBJECT: FLAME BARRIER CHARACTERISTICS OF TEXTILE FIBERS
INVESTIGATOR: Edward L. McLeod
ABSTRACT: Methods of sample preparation and testing were developed to determine the relative performance of various textile fibers as impediments to applied flame. Tests were conducted on randomly oriented compressed fiber pads.

It was found that residual products of ash-forming fibers continued to obstruct applied flame which temperatures detected on the opposing side increased at a varying rate with time. Melting and non-ash forming fibers receded from applied flame without an increase in detected temperature until the point of complete flame penetration. In some instances, nylon 66 showed some departure from the normal pattern of other melt type fibers. It was established that increased weight resulted in increased temperature lag time while the most favorable density for each fiber occurred at some intermediate degree of concentration.

WADD TR 60-510, OTS Release

October 1960

SUBJECT: DETERIORATION OF TEXTILE MATERIALS BY ULTRAVIOLET LIGHT
INVESTIGATOR: William S. Wilcox, Charles V. Stephenson, James C. Lacey, Jr.,
Bobby C. Moses
CONTRACT: AF 33(616)-6565, Southern Research Institute
ABSTRACT: This is the report on a project to determine the effects of ultraviolet radiation on polyethylene, Teflon, nylon, Mylar, and Acrilan. These materials have been irradiated in a vacuum and in an inert atmosphere of nitrogen.

Deterioration of these materials is faster for irradiation with short wavelength ultraviolet than it is for long wavelength radiation. Deterioration of polyethylene, nylon, Mylar, and Acrilan is faster in a nitrogen atmosphere than it is in a vacuum. However, Teflon deteriorates faster in a vacuum.

Experimental evidence as to the processes of deterioration are discussed in this report and mechanisms are proposed by which deterioration could take place.

WADC TR 53-373, Sup 8

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September 1960

SUBJECT: INVESTIGATION OF THE HIGH SPEED IMPACT BEHAVIOR OF FIBROUS MATERIALS PART I. Design and Apparatus

INVESTIGATOR: Chauncey C. Chu, Robert J. Coskren, Henry M. Morgan

CONTRACT: AF 33(616)-6321, Fabric Research Laboratories, Inc.

ABSTRACT: A high speed impact test machine has been designed, constructed, and calibrated to test parachute components at high rates of loading. This instrument is capable of rupturing materials of up to 10,000 pounds static breaking strength at velocities of from 200 to 750 feet per second. The impacting force is applied by a free flying missile launched by a gas gun utilizing either nitrogen or helium gas at moderately low pressures. The gun has a bore of 2.5 inches and fires missiles weighing up to 10 pounds.

Pertinent data are obtained by means of multiple exposure photography using a multi-microflash lighting source which provides a maximum of fifteen separate flashes spaced at predetermined intervals of between 10 and 10,000 microseconds. The resulting photograph records the specimen and the impacting missile before, during and after the impact. Measurement of the distances between successive exposures yields information such as the breaking strength, the extension to rupture, and the energy absorbed by the specimen.

FUELS, LUBRICANTS AND FLUIDS

FUELS

WADD TR 60-461, OTS Release

September 1960

SUBJECT: AN ANALYZER FOR MOISTURE AND SOLIDS IN JET FUELS
INVESTIGATOR: Michael Czuha, Jr., Kenneth W. Gardiner
CONTRACT: AF 33(616)-6588, Consolidated Electrodynamics Corporation
ABSTRACT: An instrument for continuously and simultaneously measuring total water, suspended water, and dissolved water in jet fuels was developed in conjunction with the anti-icing program at WADD. Studies were also made of photometric techniques for solid contamination detection and of the adaptability of a nephelometric method to the moisture analysis equipment to form a unitized contaminants analyzer.

The continuous coulometric analysis of total and dissolved water in JP-4 and RP-1 fuels furnished by WADD was effected over several ranges in a dual analyzer and the suspended water was determined by the difference in the two readings. The instrument response was correlated with Karl Fischer analyses using a polarized electrode end point indicator.

A positive interference with the instrument reading was observed with methyl carbitol in the fuel as an anti-icing additive used in early studies at WADD.

An optical system utilizing forward scattering of light and multiplier phototubes in a ratio measuring circuit was incorporated into the instrument to detect solid contaminants in the range 0-10 ppm. The response of the analyzer was determined by adding test dust to the fuel and correlating with gravimetric determinations using Millipore filters.

WADD TR 60-908

January 1961

SUBJECT: SERVICE TEST EVALUATION OF FILTERABILITY AND WATER SEPARATION CHARACTERISTICS OF JET FUEL VOLUME I AND II
INVESTIGATOR: Robert K. Johnston, J. P. Cuellar, Jr.
CONTRACT: AF 33(600)-39425, Southwest Research Institute
ABSTRACT: A study was made to determine to what extent fuel corrosion inhibitors interfere with fuel filtration and water separation. Performance was rated on contaminated JP-4 fuel in a single-element filter-separator test facility, using filter housings designed for each of five commercial element types to simulate flow conditions in the corresponding full-scale units. Standard coarse A/C test dust and water were used as contaminants; a brief study was also made using iron oxides.

WADD TR 60-908 (Continued)

The corrosion inhibitors interfered with filtration and coalescence, the effects ranging from slight to very severe. The various element types differed in efficiency and in modes of failure. Fuel aromatics content and gravity, within the JP-4 range, had little effect on performance.

The CRC water separometer, a bench-scale coalescing test apparatus, gave a general correlation with the single-element results. Interaction of additives with fuel components caused severe repeatability problems in early work, but apart from this the separometer repeatability was satisfactory. Preliminary work on a separometer reference fluid indicated that an "odorless solvent" (alkylate) may be suitable.

HYDRAULIC FLUIDS

WADD TR 59-592

May 1960

SUBJECT: GAMMA IRRADIATION OF AN ELECTRO-HYDRAULIC SERVO TEST LOOP
USING ORONITE 8515 HYDRAULIC FLUID
INVESTIGATOR: A. MacCullen
CONTRACT: AF 33(600)-32055, Lockheed Nuclear Products
ABSTRACT: This summary technical report of the irradiation of an electro-hydraulic servo test loop dynamically operated in a gamma environment includes a brief historical background, the object and scope of the program, and the procedures and results. The test was conducted for 260 hours with an average dose rate of 1.1×10^7 ergs/gm-hr (C). The fluid temperature was maintained at 275°F, and the nominal pressure was 3000 psi.

WADC TR 59-760

April 1960

SUBJECT: HIGH TEMPERATURE FLUID EVALUATION TEST STAND
INVESTIGATOR: Luke J. Gabrovic
CONTRACT: AF 33(616)-3126, Sundstrand Aviation Division
ABSTRACT: A hydraulic fluid test stand with the capability of evaluating fluids at temperatures up to 700°F and pressures up to 3000 psi, has been constructed. A survey of the industry showed several areas in which reliable aircraft type components were not available, and in such instances, the best non-aircraft components were adapted to complete the test stand.

The report describes the fluid evaluation parameters, the finalized test procedures to be used for fluid analysis, and the selection of components.

The test results of two fluid evaluations are included in the report.

WADD TR 60-250, Part I, OTS Release

May 1960

SUBJECT: HIGH TEMPERATURE HYDRAULIC FLUIDS - PART I. Chemical Degradation in a High Temperature, High Pressure Circulation Loop

INVESTIGATOR: Robert J. Benzing, Leslie R. Drane, Jr.

ABSTRACT: A study was made to observe the nature and intensity of chemical degradation resulting from severe mechanical shearing stresses at elevated (400°F) temperature. The degradation of these four fluids (two silicones, a silane, and an alkoxy disiloxane) were also studied under static thermal conditions. The physical and chemical properties, particularly viscosity, neutralization number, and flash point showed changes in direction of values with time. Possible mechanisms of break-down to explain observed differences between the static and dynamic tests are discussed.

WADD TR 60-352, OTS Release

June 1960

SUBJECT: PERFORMANCE OF PETROLEUM BASED HYDRAULIC FLUIDS IN A PUMP LOOP AT 3000 PSI AND 275°F

INVESTIGATOR: Robert N. Johnson

ABSTRACT: Fluid breakdown and pump wear were investigated in a pump loop at 275°F and 3000 psi on five petroleum based hydraulic fluids.

The mechanical shearing of these fluids resulted in excessive loss of viscosity, although their resistance to oxidation was not appreciably changed. The wide variations in the wear data of these fluids indicated their difference in lubrication characteristics in the particular system employed. Bench wear data did not correlate with the pump wear results.

WADD TR 60-394

July 1960

SUBJECT: INVESTIGATION OF THE LUBRICATING CHARACTERISTICS OF HIGH TEMPERATURE POWER TRANSMISSION FLUIDS

INVESTIGATOR: Budd N. Barclay

CONTRACT: AF 33(616)-6498, Vickers Incorporated

ABSTRACT: This program was conducted to test and determine the feasibility and potential of high temperature power transmission fluids for use in 550°F hydraulic systems. These fluids were tested for a particular purpose, and the results do not reflect on the ability of the fluids to operate satisfactorily under other conditions. The following fluids were tested under contract AF 33(616)-6398:

1. Mineral Oil MLO 7460 - A superrefined mineral oil.
2. Silicone MLO 59-250 - A halogenated methyl phenyl silicone.
3. Silane MLO 59-92 - Diphenyl d-n-dodecyl silane.

WADD TR 60-394 (Continued)

4. Silane Mixture - 50% Silane MLO 59-92 and 50% Silane MLO 59-91 (MLO 59-91 is essentially the same as MLO 59-92, but contains a lesser quantity of viscosity index improver).

5. TMP Ester MLO 59-699 - ester derivative of trimethanol propane.

6. Silicone-Ester Blend MLO 59-700 - 50% TMP ester MLO 59-699 and 50% chlorinated phenyl methyl silicone.

7. M-Bis (m-Phenoxyphenoxy) Benzene MLO 59-692.

The test fluids were subjected to 550°F testing at 3000 psi in a Vickers axial-piston pump designed for high temperature operation. Of the fluids tested, the silane-base formulations, Silane MLO 59-92 and Silane Mixture (MLO 59-92 and MLO 59-91), exhibited the best apparent potential for 550°F operation, enabling pump runs of 50 and 100 hours respectively.

The low viscosity of MLO 7460 at 550°F resulted in pump bearing failures; nevertheless, this fluid appears to offer a good potential for high temperature applications. The MLO 59-692 fluid, m-Bis(m-Phenoxyphenoxy) Benzene, exhibited excellent thermal stability, but the 550°F viscosity was too low to enable continued operation of the test pump.

Silicone MLO 59-250, TMP Ester MLO 59-699, and Silicone-Ester Blend MLO 59-700 did not maintain adequate chemical stability at 550°F. The consequent increase in acid content resulted in pump damage and necessitated early termination of tests.

The results of this program indicate that operation of a hydraulic pump on existing hydraulic fluids at 550°F is feasible. It is further concluded that the present high temperature pump design can be modified to enable extended operation on some of the fluids tested.

WADD TR 60-467

July 1960

SUBJECT: SHEAR STABILITY OF HYDRAULIC FLUIDS BY SONIC SHEAR

INVESTIGATOR: Donald C. Trop

ABSTRACT: This report presents conclusive proof that shear stability data obtained on hydraulic fluids by sonic irradiation compares quite favorably with data obtained by the present shear stability test procedure (pump-testing) in the MIL-H-5606 Specification. The sonic irradiation method should be used to replace the present pump testing procedure since it reduces test time from hours to minutes and sample quantities from gallons to milliliters.

WADD TR 60-583, Part I

September 1960

SUBJECT: LUBRICITY OF EXPERIMENTAL HIGH TEMPERATURE POWER TRANSMISSION FLUIDS - PART I. Pump Wear at 275°F
INVESTIGATOR: Leslie R. Drane, Jr., R. J. Benzing
ABSTRACT: The lubricity of several experimental high temperature power transmission fluids of widely varying chemical types was investigated. A New York Air Brake Company pump was used at 275°F as a means of studying the wear behavior. Four-Ball wear tests also were run. The fluids consisted of a silicone-ester blend, and ether and two disiloxanes. In general, these high temperature fluids gave comparable lubrication to that afforded by normal temperature petroleum fluids. In most cases the desired five hundred hours of operation were successfully completed. Such performance demonstrates the feasibility of the experimental fluids. No correlation could be obtained between the Four-Ball wear tests and the pump wear tests.

WADD TR 60-795, Part I, OTS Release

December 1960

SUBJECT: EXTREME TEMPERATURE RANGE ORGANIC COOLANTS - PART I. -80°F to 400°F Temperature Range Fluids
INVESTIGATOR: Lt Dale A. Barsness
ABSTRACT: The increased performance demands of advanced aerospace systems dictate the requirement for new and improved heat transfer materials capable of operating over extreme temperature ranges. A review of the status of extreme temperature range (ETR) coolants for electronic equipment is presented. The fluids considered for use over a -80°F to +400°F temperature range are described in this report and fall into the following class of compounds: amines, chlorofluoro-carbon oils, fluoroochemicals, silicones, silicate esters, and siloxanes. Results show that the silicone fluids comply most closely to the physical properties required for ETR electronic coolants. It was observed that they are the only group of materials that possess high boiling points and thermal stabilities above the 400°F bulk operational temperature, and still display reasonably low viscosity values within the extreme temperature range. The electrical properties of these materials are also very desirable.

The data on the new experimental research fluids, reported herein, list properties important to future coolants for electronic components. These data include the viscosity-temperature relation, vapor pressure-temperature properties electrical characteristics, thermal properties and flammability characteristics.

WADD TR 60-855

January 1961

SUBJECT: LUBRICATION BEHAVIOR AND CHEMICAL DEGRADATION CHARACTERISTICS OF EXPERIMENTAL HIGH TEMPERATURE FLUIDS AND LUBRICANTS
INVESTIGATOR: Vernice Hopkins, Andrew D. St. John
CONTRACT: AF 33(616)-6854, Midwest Research Institute

WADD TR 60-855 (Continued)

ABSTRACT: The results of seven 100-hr fluid shear stability experiments, six at 550°F and one at 275°F, indicate that MLO 56-834 (chlorinated phenyl silicone), and MLO 57-637 (di-n-dodecyldiphenylsilane) resist degradation from high shear stresses. MLO 59-98 (50-50 blend of phenyl methyl silicone and ester of TMP), and MLO 8200 (hexa-2-ethyl butoxydisiloxane) experienced a molecular change which substantially altered their properties. MLO 7277 (super-fined mineral oil) experienced a slight molecular change, and its properties were slightly changed. An experiment is described where the wear of various lubricated contacts can also be studied along with the shear stability of experimental fluids.

An apparatus for studying lubricant behavior in rolling and rolling-sliding contacts is described and results for initial runs are presented. The initial wear-life results of solid film lubrication of spherical surfaces are presented and discussed. Based on data from a plane on a cylinder configuration, these results were lower than expected. Steps taken to make the high pressure viscometer a self-contained working unit are outlined and the pressure limitation resulting from bellows configuration and fluid compressibility is discussed.

Results for extreme pressure behavior of lubricants are presented for a series of runs at 600°F and 275°F. Mating surfaces of M-10 tool steel were used for the 600°F runs while 52100 steel was used for the 275°F.

HEAT TRANSFER FLUIDS

WADD TR 60-303, Part I, OTS Release

August 1960

SUBJECT: THE EFFECT OF GRAVITY ON FREE CONVECTION HEAT TRANSFER -
THE FEASIBILITY OF USING AN ELECTROMAGNETIC BODY FORCE

INVESTIGATOR: David A. Kirk

ABSTRACT: Theories concerning the instability and motion of a fluid heated from below predicts the effect of various physical parameters. Experimental investigations have succeeded in widely varying all parameters, except gravity, in order to determine the validity of the theories. In extra-terrestrial heat transfer applications, such as in free convection, boiling, and condensation, the effect of gravity should be verified by experiment.

This report considers the feasibility of using an impressed electromagnetic body force acting parallel to the earth's gravitational field. The resultant of this electromagnetic force and the ground level gravitational force is said to be an "equivalent gravitational" force acting on the fluid mass. Based on the analysis made, a series of experiments can be performed to ascertain the quantitative contribution of gravity, over the range from -13.6 to 15.6 times ground level gravity, on free convection heat transfer. An apparatus is presently being fabricated to conduct an experiment for the case when heat is applied from below.

LUBRICANTS

WADC TR 55-30, Part VIII

June 1960

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS
INVESTIGATOR: E. Erwin Klaus, Merrell R. Fenske, Elmer J. Tewksbury
CONTRACT: AF 33(616)-5460, Pennsylvania State University
ABSTRACT: This report describes work carried out on a continuing program to characterize the capabilities of various base stocks and fluid formulations for application as hydraulic fluids and/or jet engine lubricants under a wide variety of conditions. An antiwear or chemical polishing mechanism similar to that suggested for tricresyl phosphate is shown for silicon-containing fluids with ferrous bearing surfaces. Variables designed to improve yield, efficiency, and fluid properties are discussed for low temperature solvent dewaxing. In many cases, the viscosity-temperature characteristics of the oil and wax fractions are both better than those of the original oil prior to separation. Formulation, distribution, and hardware testing of several mineral oil fluids for use over the range of -65° to $>700^{\circ}\text{F}$ are noted. Results obtained in a 550°F vickers pump test program are discussed. The basic trends in thermal degradation with mineral oils have been studied in a pressure cylinder. Quantitative evaluation of the gaseous and liquid reaction products suggest different mechanisms for the gas and liquid phase reactions. The effectiveness of inhibitor combinations as antioxidants versus a single inhibitor is illustrated at temperatures of 347° , 400° , and 500°F . The use of a small volume test to evaluate materials available in limited quantities is discussed. A series of samples which have been in storage for periods of 2 to 17 years are being reevaluated for changes in properties due to storage. An insight into the mechanism of oxidation on or in the vicinity of hot metal surfaces is provided by quantitative oxidation studies in the controlled atmosphere panel coker. A series of successive tests in the panel cokers has been used to explore further the mechanism of coking and the variables governing the deposit. Deposition type tests in a single-pass lube rig are also discussed.

WADC TR 55-240, Part VI

April 1960

SUBJECT: BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES
INVESTIGATOR: John J. Chessick, Albert C. Zettlemoyer, James F. Wightman, Lovella Raub
CONTRACT: AF 33(616)-3999, Lehigh University
ABSTRACT: A systematization of the most probable states for solids dispersed in organic media has been made. Specific attention has been paid to the development of more rigorous definition where ambiguity existed previously. Flocculation is discussed in terms of (primary) van der Waals type attractive forces and secondary and tertiary attractive forces. These latter two result from the influence of flocculating agents. Reduced deflocculation occurs due to steric hindrance, adsorbed lyosphere, entropic repulsion or double layer effects.

WADC TR 55-240, Part VI (Continued)

An investigation designed to determine the causes of structural instability of non-soap greases during use, and the application of the knowledge obtained to the production of superior greases was initiated. Various techniques such as ion incorporation or resin treatment of thickener solids were employed to increase the mechanical and temperature stability of greases containing various inorganic thickeners. Good success was achieved here, apparently. Grease with excellent stability properties can be prepared with surface-modified thickeners. Surface modification was accomplished by deposition of phenol-formaldehyde type resins onto the surfaces of the solids in question followed by curing at elevated temperatures to a stable, coherent, though not necessarily, a monomolecular film.

Miscellaneous work included the construction of a high temperature Shell Roll Tester and limited studies of the influence of additives on grease structure.

WADC TR 57-299, Part III

April 1960

SUBJECT: NUCLEAR RADIATION RESISTANT LUBRICANTS

INVESTIGATOR: William L. R. Rice, David A. Kirk

ABSTRACT: This report is intended to present a summary of the state-of-the-art of extreme temperature, nuclear radiation resistant fluid and lubricant development. It presents data on the effects of gamma radiation on a number of commercial fluids and lubricants, with a discussion of potential problem areas. Selection of improved materials of greatly increased thermal and nuclear radiation stability is discussed and the present status of preferred lubricants is outlined.

It is estimated that on a basis of information to date, the following conclusions can be made:

1. That up to the dosages (2.6×10^{11} ergs/gm carbon gamma and 3×10^{16} nvt fast neutrons) investigated ambient temperature wear life of commercial solid film lubricants did not show significant changes on irradiation.

2. Data on the silicone base greases show that the large changes which do occur in dropping point, bomb oxidation, bomb copper corrosion, and oil separation had little to no effect on the bearing performance tests. Preliminary in-source bearing tests on an arylurea silicone blend have performed satisfactorily for an average of 500 hours at 450°F and at a dose rate of 4.5×10^7 ergs/gm carbon hour.

3. The most promising candidate high temperature radiation resistant engine oils are the polyphenyl ethers. Laboratory tests indicate that they should be useable to bulk oil temperatures as high as 600°F in the presence of integrated nuclear dosages of 1×10^{11} ergs/gm carbon.

WADC TR 57-299, Part III (Continued)

4. In-source testing of a hydraulic loop showed that failure of Viton A dynamic seals could be expected at 3.0×10^9 ergs/gm carbon gamma dosage and at 275°F.

5. Aircraft instrument oils (MIL-L-6085) oxidation-corrosion stability was severely diminished at gamma dosages as low as 8.7×10^8 ergs/gm carbon.

WADC TR 58-297, Part III

April 1960

SUBJECT: DEVELOPMENT OF EVALUATION TECHNIQUES FOR DETERMINATION OF THE LUBRICITY AND STABILITY OF NEW HIGH-TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS

INVESTIGATOR: Vernice Hopkins, Andrew D. St. John

CONTRACT: AF 33(616)-5202, Midwest Research Institute

ABSTRACT: An investigation has been conducted to determine the initial pumping rates of seven synthetic lubricants and one super-refined mineral oil at temperatures down to -95°F. Most lubricants were pumped at temperatures below their pour points. Peak torques experienced during a simulated cold start were many times the torque required at rated conditions.

A high-temperature hydraulic fluid pump test stand has been set up, its performance evaluated at temperatures up to 700°F, and pressures up to 3,000 psig. Test results are presented for three 100-hr. tests, two at 400°F, and one at 550°F. Changes are described which were made to improve the pump stand's performance and reliability.

A bearing analysis has been completed which indicates that the energy dissipation and coolant roles of lubricant in the bearing depend on average metal body temperature; the two roles are otherwise isolated from one another. The design of a tester is outlined which separately measures the lubricant dissipation and cooling properties.

A four-ball wear tester has been modified for 1000°F operation. Tests have been run on a group of experimental lubricants at temperatures up to 1000°F using a special steel and M-10 tool steel. These tests were conducted to see if a sliding contact of special steel was better lubricated than M-10 tool steel with this group of lubricants. The M-10 tool steel was generally worn the least except when lubricated with a silicone.

Tests have been run on the WADD E. P. tester to evaluate its performance and to obtain information for establishing a fluid evaluation procedure. A tentative procedure for seizure load determination has been written. Seizure load data are presented for a di-ester and an E. P. lubricant with a 250°F lubricant temperature. When 52100 steel test elements are used, the seizure load for the E. P. lubricant is greater than that for the di-ester; however, when M-10 tool steel test elements are used, seizure was not experienced with either lubricant.

WADC TR 58-297, Part III (Continued)

A high-pressure viscometer capable of operating at temperatures up to 400°F and pressures up to 250,000 psig has been set up in the lubrication laboratory at the MRI Deramus Field Station. Seal problems prevented the evaluation of its performance. Steps have been taken to correct the seal problems and to make it independent of MRI capital laboratory equipment.

An oscillatory bearing tester has been modified for 1000°F operation. It is planned to use this machine to evaluate dry film lubricants applied to a spherical ball bearing.

An environmental chamber system has been completed in which accessory components can be mounted and used as test rigs for the practical evaluations of lubricants and power transmission fluids. The system consists of a -100 to 800°F environmental chamber, a 50 h.p. 0-12,000 rpm variable speed drive, 28 v. DC and 208v., 400 cycle power supplies, and controls and instrumentation.

Specifications have been written for a -300 to 1500°F, 10^{-5} mm of Hg "walk-in" altitude chamber and a 25 h.p. 50,000 rpm variable speed drive.

WADC TR 58-350, Part III

June 1960

SUBJECT: DEVELOPMENT OF GREASES FOR HIGH SPEED BALL AND ROLLER BEARINGS
INVESTIGATOR: Paul R. McCarthy, Guy C. Blewett, John F. Hedenburg
CONTRACT: AF 33(616)-5020, Gulf Research and Development Company
ABSTRACT: During this phase of the contract, approximately forty fluids and ninety experimental thickeners were evaluated as components for greases intended for operation at 20,000 rpm and 400°F.

The most promising of the fluids were certain of the silicones, m-bis(m-phenoxyphenoxy)benzene and a silphenylene. Since the m-bis(m-phenoxyphenoxy)benzene and silphenylene were in short supply, very limited work was done with these fluids.

Of the thickeners used primarily in conjunction with DC-550, certain lots of spiro(hydantoin-5,1'-indan) and 1,1'-methylenebis(5,5-dimethylhydantoin) produced experimental greases which operated satisfactorily for a period of 500 hours at 400°F and 20,000 rpm. Greases prepared with other lots of the same materials failed in much shorter periods. Arylureas and combinations of arylureas and Benzidine Yellow as thickeners may provide greases showing even better performance under the above conditions. More extensive evaluation is needed to substantiate this.

Statistical analyses to determine (a) correlation between screening tests and performance of greases in antifriction bearings and (b) precision limits on performance life tests at 20,000 rpm and 400°F were made. Results on the analyses indicated no correlation for (a) and very poor precision for (b).

WADC TR 58-350, Part III (Continued)

Construction of two 45,000 rpm extreme high speed testers was completed and one of these testers put into operation. The second tester will be in operation by February 10, 1960. Operation of the tester and accessory control equipment is very good. Sixteen trial runs on two greases showed very short life (0.25 to 10 hours) for the greases and bearings at speeds above 30,000 rpm.

WADC TR 59-633, Part II

December 1960

SUBJECT: HIGH TEMPERATURE INSTRUMENT OIL
INVESTIGATOR: Albert A. Schwartz, Harry R. Broadley, Jr., Robert S. Norman
CONTRACT: AF 33(616)-5894, General Electric Company
ABSTRACT: Studies were made on lubricity improvers and oxidation inhibitors for silicone fluids, which had been found in our earlier work to be the most promising base stocks. Preliminary screening of the various fluid additive combinations were carried out using procedures developed in Part I of the program. Full scale performance tests in instrument bearings were run in air and in argon atmosphere. Significant improvement in performance in both the preliminary screening and the bearing tests was achieved with several of the lubricity additives. A vendor-developed dry lubricated ball bearing looked very promising in the bearing life tests.

Evaluation of the base stock, the most promising fluid formulation, and the vendor-developed dry lubricated ball bearing was carried out in a simulated gyro gimbal assembly and in high speed gyro motors designed to operate at 400°F. Significant improvement over the silicone base oil was obtained with the oil compounded with a lubricity improver, especially in the slow speed gimbal tests. Lubrication of the high speed gyro motors with these lubricants was not as satisfactory as the slower speed application.

The dry lubricated bearings developed by the outside vendor appear promising for moderate to high speed applications, such as the tachometer generator, where torque requirements are not critical. These bearings did not prove to be satisfactory in the gyro gimbal test since bearing torque increased rapidly during the first 200 hours of operation.

WADC TR 59-736, Part II, OTS Release

May 1960

SUBJECT: LOAD SUPPORT AND ANTI-SCUFFING PROPERTIES OF LUBRICANTS
PART II. WADC Universal Gear and Spline Lubricant Tester
INVESTIGATOR: Leslie R. Drane, Jr.
ABSTRACT: The design and operating characteristics of a gear and spline lubricant tester are described. Results of studies to determine the effects of lubricant flow rate, gear speed, and inert atmosphere on gear scuff load are presented.

WADC TR 59-736, Part II (Continued)

Lubricant load carrying ability decreased sharply in the 5000 to 10,000 rpm range, but leveled off in the 15,000 to 20,000 rpm range. A slight increase in scuff load with increasing speed was noted in some cases.

The use of an inert atmosphere caused a decrease in scuff load rating with a mineral oil, a sebacate, and a silicone-ester blend. Two silicone fluids showed no significant change in scuff load when tested in an inert atmosphere.

WADD TR 60-424

November 1960

SUBJECT: HIGH TEMPERATURE LUBRICATION IN THE PRESENCE OF NUCLEAR RADIATION
INVESTIGATOR: V. N. Borsoff, S. J. Beaubien, W. W. Kerlin
CONTRACT: AF 33(616)-6658, Shell Development Company
ABSTRACT: The work presented is an investigation of lubricating capabilities of unsubstituted polyphenyl ethers and a comparison of these ethers with conventional lubricants. The work consists of studying the performance of the lubricants in bearings and gears under severe thermal, oxidative and ionizing radiation stresses. Based on the evidence obtained, it appeared that all lubricants suffer an appreciable decrease in load carrying capacity at elevated temperatures, but preserve their lubricating properties under the most severe environments, providing the flow of oil to the load bearing elements is not impeded in any manner. Since the flow of oil is highly affected by degradation due to heat, oxidation and ionizing radiation, the stabilities of unsubstituted polyphenyl ethers and MIL-L-9236A oil were studied. The results showed a great superiority of the polyphenyl ethers over the conventional oils. Regarding the effect of ionizing radiation, it was noted that at levels below 1×10^9 ergs/g C its effect is negligibly small and in this radiation range the main causes of lubricant degradation are heat and oxidation.

WADD TR 60-529

October 1960

SUBJECT: MECHANISM OF LUBRICATION OF GRAPHITE SINGLE CRYSTALS
INVESTIGATOR: Paul J. Bryant
CONTRACT: AF 33(616)-6277, Midwest Research Institute
ABSTRACT: A research program has been initiated to investigate the friction mechanism of graphite. The effects of atmosphere, crystal orientation, etc., on the lubricative process are being observed.

A search for satisfactory graphite samples has been undertaken with graphite whiskers being considered first. The whisker structure was first found to be imperfect when it was physically removed from the growth matrix. A technique of selective oxidation was developed to extract the whiskers

WADD TR 60-529 (Continued)

from the matrix without physical damage. Whiskers thus extracted, while still not ideal, are useful in some phases of the study. In particular, they contain a uniform inner core which is useful for surface friction, atmosphere, and wear damage studies.

Single crystals of graphite were produced by the thermal decomposition of aluminum carbide single crystals. These samples have the regular graphite crystalline orientations throughout, and are representative of the fundamental graphite system.

Aluminum-oxide whiskers were grown as a by-product of the technique for producing graphite single crystals. Their structure and the mechanism by which they were produced are of interest because they indicate the temperature conditions which existed during the production of graphite crystals.

X-ray diffraction analysis of the single crystals showed uniform crystallographic orientations and interatomic spacings in close agreement with those reported for natural graphite. The graphite whiskers show normal graphite spacing, but their crystal orientations were not uniform. The aluminum-oxide whiskers were found to consist entirely of the alpha phase and to contain no measurable impurities.

Structural features of the graphite whiskers and single crystals as determined by optical microscopy and induced damage experiments indicate the type of experiments for which the whiskers and single crystals are suitable.

WADD TR 60-530

September 1960

SUBJECT: CERAMIC BONDED SOLID-FILM LUBRICANTS
INVESTIGATOR: M. T. Lavik
CONTRACT: AF 33(616)-6115, Midwest Research Institute
ABSTRACT: Thirty-three compounds were screened for their oxidation resistance in air. Sixteen compounds indicate less than 50 percent conversion to oxides after 4 hr. at 1000°F. The same compounds were screened for frictional properties from 80° to 1000°F at light loads. Seven of them indicated a friction coefficient below 0.50 over a large portion of the temperature range.

Four high temperature bonding agents were selected for bonding films of the most promising solid lubricants. Outstanding results were obtained at 1000°F with PbS films bonded with B_2O_3 . Wear-lives as high as 50,000 revolutions were recorded at 1000°F, 100-lb. load and 370 rpm, with friction coefficients from 0.07 to 0.15 and negligible wear on the rub shoes. The wear-life of the PbS/ B_2O_3 films falls off very rapidly with decreasing temperature. Films combining MoS_2 or graphite with PbS/ B_2O_3 films have not shown good results at 1000°F.

WADD TR 60-536

November 1960

SUBJECT: HIGH TEMPERATURE LUBRICANTS FOR ADVANCED GAS TURBINES
INVESTIGATOR: Harold W. Adams
ABSTRACT: Selected polyesters derived from trimethylol propane proved to be satisfactory base stock fluids from -65°F to bulk oil temperatures of 400°F to 450°F , for advanced gas turbine engines. A combination "inhibitor package" consisting of 5-10-10 diphenylphenazasilane and phenyl alpha naphthyl amine, was found to be most effective in these trimethylol propane fluids. The finished fluid formulations (resulting from the above base fluids and additive combinations) provided the necessary physical and chemical characteristics required for advanced gas turbine lubricants.

WADD TR 60-557, Part I

October 1960

SUBJECT: DEVELOPMENT OF GREASE LUBRICANTS FOR HIGH TEMPERATURE BALL AND ROLLER BEARINGS OF ELECTRICAL EQUIPMENT
INVESTIGATOR: A. C. Borg, K. R. Bunting, A. M. Dobry, J. C. Goossens, J. H. Klauwers, R. S. Barnes
CONTRACT: AF 33(616)-6584, Standard Oil Company
ABSTRACT: The objective of this contract is the development of grease systems for use in ball and roller bearings of electrical equipment over an ultimate temperature range of -65°F to 900°F . The current program is aimed at the development of a -40°F to 700°F grease. The high temperature performance is the opening and screening fluids, thickeners and greases at 600°F . 600°F was chosen because long time performance at this temperature is required by the contract and it was close to the limit of significantly long performance of available greases and test equipment at the beginning of the contract.

Equipment was built or obtained for running loaded bearing tests, dropping points, evaporation, and roll stabilities at temperatures up to 900°F . Several types of bearings, said to be better for high temperature use than the MRC 204 S-17 bearings used in this period, were received at the end of this reporting period, but no tests have yet been run on them.

Fluids and thickeners, believed to be potentially useful in -40°F to 700°F greases, were synthesized or obtained from outside sources and screened in bench tests for adequately high thermal stability and low volatility. Thickeners were also screened for thickening ability over the temperature range. Materials that appeared promising after screening were made into greases and bearing tested. Fluids were thickened with the best high-temperature thickener available, and thickeners were dispersed in the best high-temperature fluid to obtain the greases for these tests.

Most bearing tests were run at 600°F , under modified CRC L-35 conditions, on Pope-Texas type spindles. Under these conditions, the best of the currently available greases, methylphenyl silicones, thickened with one of several high-melting organic thickeners ran for about 55 hours.

WADD TR 60-557, Part I (Continued)

Greases made with several experimental silicone fluids gave results that ranged from slightly worse to significantly better than those obtained with the best available greases. The best results were obtained with DC-QF-6-7024 Silicone Fluid which ran from 100 to 400 hours in various tests. Failures were due to the instability of the silicone fluids. The best phenoxyphenyl ether and chain-type polyphenyl greases ran as long as the best of the currently available greases. Failures were not due to fluid instability, but fluid volatility. Preparation of lower volatility fluids of these and related structures are under way. Surface treated silica, carbon black and glass fiber thickeners gave better results than the best of the currently used high temperature thickeners. In addition, some promising initial results were obtained with high melting polymers, complex sulfonyls and sulfonamides, and some organic pigments. None of these thickener systems has been completely perfected and even better results are to be expected as they are further developed.

Running similar bearing tests on mRC spindles gave results equivalent to those obtained on the Pope-Texas spindles, when similar load conditions were used. Under higher load conditions, shorter test runs were used. The DC-QF-6-7024 Fluid grease ran only 8 hours when the axial load was increased from five to fifty pounds. Load carrying additives improved bearing performance at higher loads. One additive in the QF-6-7024 grease, lowered the five pound axial load running time to about 60 hours, but raised the fifty pound running time to over 30 hours. Another additive when added in this grease, did not affect the five pound load life, and ran for over 40 hours at fifty pounds load.

Increasing the temperature to 650°F decreased the running time.

Increasing the amount of grease filled into the bearing increased the running time.

No tests were run with relubrication, but the development and use of such a technique coupled with the use of better bearings is expected to increase bearing running times.

WADD TR 60-757

December 1960

SUBJECT: SILICONE LUBRICANT-CARBON SEAL INTERACTION AT ELEVATED TEMPERATURES
INVESTIGATOR: Thomas H. Koenig, E. James Vargo
CONTRACT: AF 33(616)-6714, Cleveland Graphite Bronze
ABSTRACT: The advent of higher power jet engines necessitated a suitable high temperature lubricant. Silicone oils became attractive candidates for this field primarily because of their thermal stability at high temperatures.

WADD TR 60-757 (Continued)

However, one problem presented itself in the form of deposits at carbon seal areas. It was originally theorized that these deposits were formed by a basic incompatibility between silicone oil and the carbon used for mechanical seals.

Static immersion tests using 18 grades of carbon in combination with three silicone oils, one ester, and one silicone-ester blend at bath temperatures of 400°F, 500°F gave no indication that carbon has an effect on viscosity increase or high temperature breakdown of the oils tested. In tests where radical changes occurred in samples containing carbon, there were also radical changes in control samples not containing carbon. This point was also confirmed in dynamic tests.

In a test setup simulating sealing conditions, silicone sludges were formed at indicated interface temperatures of 280°F. This temperature is considerably below the oxidation and thermal decomposition temperatures of the lubricants. Localized hot spots were found to be the source of the high temperature. In one case, temperatures 300°F higher than the average were recorded.

Bench rig tests of shaft and face seals produced silicone sludge under conditions far less severe than those encountered in the field. Tests run with a nitrogen blanket show that excluding oxygen from the seal interface inhibits or stops the formation of silicone sludge. This method of seal protection will be satisfactory until the seal interface temperature reaches the thermal decomposition limit of the silicone oil, approximately 620°F.

WADD TR 60-770

December 1960

SUBJECT: SYNTHESIS AND INVESTIGATION OF HIGH TEMPERATURE GREASE THICKENERS

INVESTIGATOR: John Q. Griffith, III, John B. Christian

ABSTRACT: High melting imide solids synthesized from anthraquinone, pyromellitic acid, pyromellitic dianhydride, phthalic anhydride and various amines were investigated as potential high temperature grease thickeners.

Several commercially available high melting solids such as asbestos, resins and amine anthroquinone thickening compounds were included in this investigation.

Greases were formulated from these materials by solvent, heating, and milling techniques. Physical characteristics of the resulting greases were also determined.

An imide thickener combination comprised of two separate components, the aluminum complex of pyromellitic benzoic acid and phthalimido benzoic acid proved to be the most promising material investigated. It proved to have good thermal stability, excellent gel characteristics and a melting point above 1200°F.

WADD TR 60-770 (Continued)

The commercially obtainable high melting point solids were generally ineffective as grease thickeners. Better methods of dispersion and of keeping article size uniform would greatly facilitate this work.

SOLID FILM LUBRICANTS

WADC TR 59-127

July 1960

SUBJECT: A MOLECULAR APPROACH TO DRY FILM LUBRICATION IN A VACUUM (SPACE) ENVIRONMENT

INVESTIGATOR: R. M. VanVliet

ABSTRACT: This report presents an analysis of the problems associated with the use of dry film lubricants at moderate temperatures on unpressurized space vehicles. The effects and suspected effects of vacuum on dry film lubricants are analyzed in detail to provide the basis for future research programs in this area. Several hypotheses for lubricant environment interaction are presented and a few basic research approaches are suggested.

ELECTRICAL AND ELECTRONIC MATERIALS

WADC TR 59-337, Part II, OTS Release

September 1960

SUBJECT: SYNTHESIS AND PURIFICATION OF DIELECTRIC MATERIALS
INVESTIGATOR: W. C. Divens, D. H. Hogle, D. W. Lewis, P. A. Tierney,
T. W. Dakin, D. Berg
CONTRACT: AF 33(616)-5979, Westinghouse Electric Corporation
ABSTRACT: This report describes the progress during the second year on a research program undertaken to prepare pure dielectrics with improved properties for use as electrical insulation at 500°C. The effort has been concentrated on the materials: boron nitride, alumina, and boron phosphide.

Boron nitride has been prepared with better dielectric properties, at 500°C, than any other reported materials. The condition for the hot pressing of the boron nitride discs have been elucidated. Hot pressed boron nitride has been shown to possess electrical properties which are anisotropic.

Anodized aluminum oxide films have good 500°C dielectric properties. The films are polarity sensitive and have a resistance which decreases with increasing field.

The adhesion of the electrode metal to the anodized oxide film has been improved appreciably by first evaporating silicon monoxide in vacuum, and then evaporating gold on top of the silicon monoxide without having released the vacuum.

Dielectric measurements of aluminum oxide films formed by hydrolysis of aluminum isopropoxide have indicated inferior properties for the oxide made in this manner.

Boron phosphide has been synthesized by two means: (1) direct reaction of the elements, boron and phosphorus, and (2) by reaction of boron trichloride, phosphorus and hydrogen.

WADC TR 59-469, OTS Release

September 1960

SUBJECT: INVESTIGATION OF ORGANIC SEMICONDUCTORS
INVESTIGATOR: G. P. Brown, S. Afterbut
CONTRACT: AF 33(616)-5949, General Electric Company
ABSTRACT: The specific resistivity as a function of temperature has been determined for a variety of organic compounds including polyacrylonitrile, polyacrylonitrile containing additives, 1,1-diphenyl-2-picrylhydrazyl, naphthalene and its 1-amino, 1-nitro-, 2-methoxy-, and 2-phenyl derivatives, 1,4-diphenyl-1, 3-butadiene, 1,8-diphenyl-1,3,5,7-octatetraene, and several picrates.

WADC TR 59-469 (Continued)

The resistivities were obtained on materials in various stages of purification, over a wide range of applied voltages, in nitrogen and in air, and in the solid and (wherever feasible) in the liquid state. A zone refiner was designed and used in the purification of several materials. The results obeyed the semiconductor equation, $\rho = \rho_0 \exp(E/2kT)$; values for the energy gap E and the resistivity at infinite temperature ρ_0 were calculated. A model for conductivity in organic compounds has been advanced postulating that the primary process responsible for conductivity consists in the formation of biradicals. The pertinent literature was surveyed and the available data were tabulated.

WADD TR 60-17, OTS Release

May 1960

SUBJECT: PELTIER EFFECTS FOR CRYSTAL GROWING
INVESTIGATOR: Louis L. Thomas
CONTRACT: AF 33(616)-6150, Nuclear Corporation of America
ABSTRACT: Available literature on crystal growing and refining processes has been reviewed. In accordance with the results of the literature survey and analysis of the problem, the zone melting technique has been chosen for modification to study the potential of the Peltier effect for improving crystal properties. Zone melting apparatus, modified according to the criteria developed, has been fabricated. Germanium rods have been zone melted. Concurrent experimental work included measurement of resistivity, Hall effect, magneto-Seebeck effect at the melting point, and Peltier effect as applied to zone melting.

WADD TR 60-111, OTS Release

June 1960

SUBJECT: ORGANIC SEMICONDUCTOR STUDY
INVESTIGATOR: J. B. Rust, F. A. Haak, J. P. Nolta
CONTRACT: AF 33(616)-6024, Hughes Aircraft Company
ABSTRACT: This report covers the first fifteen months of a study on organic semiconductors under Air Force Contract AF 33(616)-6024. Experimental results for the temperature dependence of the resistivity are presented for thirteen organic compounds. Experimental results regarding the rectification properties of copper phthalocyanine and other metal derivatives of phthalocyanine are presented. A tentative theory based on the formation of a space charge due to the presence of an oxidizing agent in the system has been developed. Preparation for the study of the effects of high electric fields on anthracene and phenanthrene are discussed and some qualitative results are presented.

WADD TR 60-364, OTS Release

August 1960

SUBJECT: AN INVESTIGATION OF THE RESPONSE OF PHOTOMULTIPLIER TUBES
INVESTIGATOR: Gordon H. Griffith

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WADD TR 60-364 (Continued)

ABSTRACT: The response of a photomultiplier tube is related to its average sensitivity and can be improved by using only the most sensitive part of the photocathode. This report presents a number of graphs to illustrate the response of a photomultiplier tube as a function of the point on the photocathode surface that is illuminated and to indicate the improvement in symmetry of the iso-sensitivity lines that may be achieved by varying the shield voltage. While the observed sensitivities were due primarily to the geometry of the cathode-dynode configuration, they were also due partially to the non-uniform response of the cathode surface.

NUCLEAR MEASUREMENTS

WADC TN 59-401, OTS Release

November 1959

SUBJECT: ATTEMPT TO EVALUATE CHLORINATED HYDROCARBON DOSIMETERS BY CROSS CALIBRATING ELEVEN GAMMA RAY SOURCES
INVESTIGATOR: Robert L. Hickmott, Maurice J. Cote, Philip B. Hemmig
ABSTRACT: An evaluation of the chlorinated hydrocarbon dosimetry system of Convair, Fort Worth, was attempted by the Materials Laboratory, Wright Air Development Center, with the cooperation of a number of high intensity irradiation facilities throughout the country. The evaluation consisted of two phases. The first phase tested the precision of the dosimetry system. It involved the irradiation of thirty five single dosimeters at Wright Air Development Center. The second phase was intended to field test the dosimetry system. It was also hoped that data from this phase would permit the cross calibration of the participating facilities.

Data obtained in this program is presented with an analysis of the results. Variations from facility to facility, and within the same facility, were noted which cannot be explained by the available information, however, the program was not specifically designed to separate rate effects, spectrum effects, and temperature, storage, and handling effects which could have influenced the results.

Although the data appear inconclusive they illustrate the problems that may arise in applying such dosimetry systems, and demonstrate the desirability of further work to cross calibrate the many irradiation facilities.

WADC TR 59-443, OTS Release

September 1960

SUBJECT: FANTASIA AND TRIPROD - SHIELDING CODES FOR THE 1103A UNIVAC
INVESTIGATOR: Herbert Steinberg, Jerome Heitner, Raphael Aronson
CONTRACT: AF 33(616)-5187, TRG, Incorporated
ABSTRACT: Two codes have been developed for the 1103A Univac. FANTASIA computes neutron transmission through laminated slab shields and slowing down density within the shields by Monte Carlo methods. TRIPROD is a slowing down code suitable for shielding problems. It is based on the General Electric reactor code VALPROD. Both theory and application of FANTASIA and TRIPROD are discussed. Operating instructions are included.

WADC TR 59-607, OTS Release

May 1960

SUBJECT: STANDARDIZATION OF FABRICATION TECHNIQUES (THRESHOLD FOILS)
INVESTIGATOR: I. E. Lamb, R. R. Tsukimura
CONTRACT: AF 33(600)-35917, Aerojet-General Nucleonics

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WADC TR 59-607 (Continued)

ABSTRACT: The techniques used to develop and standardize a series of neutron detection foils for measurement of neutron spectra are described. The series includes gold and cobalt foils, and plutonium-239, uranium-238, and sulfur threshold foils. A method of calibrating the foils with a low-flux research reactor is discussed. The instrumentation required to obtain data from the foils is presented, together with the neutron intensities that may be explored with these detectors.

WADC TR 59-711, OTS Release

February 1960

SUBJECT: OPTICALLY ACTIVE ORGANIC COMPOUNDS AS HIGH LEVEL GAMMA DOSIMETERS

INVESTIGATOR: Stanley M. Dec

ABSTRACT: Materials which are of necessity located in high level radiation fields frequently undergo degrading effects. A measurement for high level radiation is an important factor in the practical application of materials in nuclear environments.

This project was initiated in order to develop a dosimeter system which could extend the range of current dosimeter methods. A new technique was used, wherein the effects on the rotation of optically active organic compounds were determined with gamma radiation. The program was directed toward obtaining characteristics of a number of probable dosimeter systems. Linear or near linear response was observed for several systems to a range of at least 10^{10} ergs gram⁻¹ carbon. Maximum range levels were not determined since a considerable length of time is needed for irradiations at higher doses.

WADD TR 60-98, OTS Release

May 1960

SUBJECT: MICROWAVE GAMMA-RAY ION CHAMBER

INVESTIGATOR: Donald A. Brown, Karl C. Speh, Jesse J. Taub

CONTRACT: AF 33(616)-5758, Airborne Instruments Laboratory

ABSTRACT: A microwave gamma-ray ion chamber and its associated equipment has been developed that is capable of measuring gamma radiation in the range from 10^6 to 10^{10} ergs per gm-hr (carbon). This equipment operates at a microwave frequency of about 9 kmc and measures radiation by using the changes in the reflection coefficient looking into a gas-filled microwave resonant cavity caused by ionization of the gas due to radiation. Data on gas conductivity and resonant frequency shift have been taken for different gas mixtures.

WADD TR 60-293, OTS Release

July 1960

SUBJECT: A PROTON-RECOIL ORGANIC SCINTILLATION SPECTROMETER AND INVESTIGATION INTO A VARIABLE THRESHOLD SPARK COUNTER

WADD TR 60-293 (Continued)

INVESTIGATOR: John B. Ashe, William E. Tucker, O. M. Hudson, Jr.,
J. T. Prud'homme
CONTRACT: AF 33(616)-6362, Texas Nuclear Corporation
ABSTRACT: This report describes the research which was conducted
by Texas Nuclear Corporation on two possible neutron spectral measuring
devices based on the detection of recoil protons.

A scintillation detector is described which has the ability to differentiate between neutrons and gamma rays by means of the different decay times of recoil proton and Compton electron scintillations. This detector has been incorporated into a neutron spectrometer which is highly insensitive to gamma rays over a detectable neutron energy region of approximately 0.8 Mev to greater than 17 Mev. Data obtained with the spectrometer are presented.

In addition to the scintillation detector, investigations into the feasibility of using a spark counter as an energy sensitive neutron detector are also described. The results of these investigations are discussed and representative data are presented.

WADD TR 60-312, OTS Release

September 1960

SUBJECT: THE SPM (STOCHASTIC PROCESS METHOD) EQUATION FOR CALCULATING
THE SPATIAL VARIATION OF NEUTRON SPECTRA
INVESTIGATOR: Carl N. Klahr, H. Julius Zell
CONTRACT: AF 33(616)-5187, TRG, Incorporated
ABSTRACT: The SPM equation is a second order partial differential equation in lethargy (or energy) and spatial coordinates. It promises to describe the slowing down and spatial diffusion of neutrons with sufficient accuracy for shielding problems and for other applications where neutron flux spectra are critical. A primitive SPM with infinite orders of derivatives is first obtained from either the Smoluchowski or Boltzmann equations. It is then truncated after the second order derivatives. The coefficients of two forms of this equation are obtained and suitable boundary conditions are given. A code has been written to solve this equation on the 1103A Univac. Various solutions are given.

WADD TR 60-344, OTS Release

November 1960

SUBJECT: A STUDY OF THE NATURE OF FREE RADICALS IN IRRADIATED
CHEMICAL SYSTEMS
INVESTIGATOR: P. Y. Feng, W. A. Glasson, S. A. Marshall
CONTRACT: AF 33(616)-6141, Armour Research Foundation
ABSTRACT: In order to better evaluate the factors which dictate the susceptibility of organic compounds to form free radicals on exposure to ionizing radiation and the nature and fate of said radicals, a study has been made

WADD TR 60-344 (Continued)

of the radiation chemistry of various organic compounds. The compounds studied were chosen in order to ascertain the effect of structure and presence of functional groups within the basic carbon skeleton on the radiation damaging of the various substrates. Compounds studied were n-, sec-, and tert-butyl iodides, tert-butyl amine, n-butanol, n-butyric acid, and ethyl acetate. The alkyl iodides were studied in order to ascertain the effect of structural isomerism on a system whose primary dissociative step is known, i.e., carbon-iodine bond fission. The other compounds were studied in order to evaluate the effect of changing the functional group (alcohol, amine, acid, etc.) and the over-all structure (ester) in a four carbon organic system.

The systems were studied using product analysis as the main tool. Gas-liquid partition chromatography was the analytical method used for the liquid products while manometric and mass spectrometric means served for the gaseous products. In addition electron spin resonance techniques were used as a means for determining directly the radical(s) present.

The results indicate that over-all yields of products increase on changing from a primary to a secondary to a tertiary bond to the dissociable entity. Further, on changing the functional group the character of the products change as the bond energy in question approaches that of the carbon-hydrogen bond. The results are in accordance with the view that the products can be predicted on the basis of the relative bond strengths within the molecule and classical theories of free radical chemistry.

WADD TR 60-544, OTS Release

August 1960

SUBJECT: RADIOCHEMICAL PRODUCTION AND FATE OF FREE RADICALS

INVESTIGATOR: Roger E. Rondeau

ABSTRACT: This report represents an effort to interpret and correlate to a limited extent, the many physico-chemical phenomena of radiation chemistry in terms of elementary free radical processes.

WADD TR 60-562, OTS Release

October 1960

SUBJECT: A SOLID STATE BRAGG-GRAY CAVITY CHAMBER

INVESTIGATOR: Victor H. Ritz, Frank H. Attix

CONTRACT: MIPR (33-616)-59-27, U. S. Naval Research Laboratory

ABSTRACT: An attempt has been made to construct the solid state analogue of a Bragg Gray cavity ionization chamber. Thin layers of materials sensitive to radiation have been placed between walls of C, Al, Cu, Sn or Pb. Two types of sensing agents have been employed; thin layers of anthracene whose ultraviolet induced luminescence degrades upon irradiation, and 6 micron thick polyethylene terephthalate (Mylar) films whose optical density at 3250A increases

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upon irradiation. Results in a Co^{60} γ -ray field with the anthracene disagree sharply with theory, while the Mylar film yields excellent agreement (+5%) with theory. A preliminary attempt to discriminate between the fast neutron and gamma ray dose in a nuclear reactor has been unsuccessful.

WADD TR 60-563, OTS Release

October 1960

SUBJECT: DOSIMETRY BY LUMINESCENCE DEGRADATION IN ORGANICS
INVESTIGATOR: Frank H. Attix
CONTRACT: MIPR (33-616)58-21, U. S. Naval Research Laboratory
ABSTRACT: This report covers an investigation of the phenomenon of photoluminescence degradation in organic materials due to damage by ionizing radiations.

A survey of previous studies of this effect is presented.

The present work stresses reduction to a practical dosimetry system for the 5×10^5 to 5×10^9 rad absorbed dose range. It is demonstrated that pressed wafers of anthracene and of p-querterphenyl can be employed together to cover the required dose range. Ultraviolet light (3650 angstrom) is used to excite the photoluminescence, which is observed in a narrow band at 4420 angstrom.

Some recovery of photoluminescence with time after X-ray irradiations is observed, and heat treatment is proposed as a method of reducing this effect.

Degradation of luminescence in biphenyl, p-terphenyl, fluorene, naphthalene, and tetracene have also been studied and are reported upon.

WADD TR 60-645, OTS Release

November 1960

SUBJECT: RADIATION EFFECTS ON ELECTRONIC COMPONENTS: A COMPARISON OF PAPER CAPACITORS IMPREGNATED WITH VITAMIN "Q" MINERAL OIL AND BIS-(P-PHOXYPHENYL) ETHER
INVESTIGATOR: Dennis R. Johnson, Milton E. L. Zellmer
ABSTRACT: Bis-(p-phenoxyphenyl) ether (BPPE) when irradiated proves to be highly resistant to radiation damage. Paper capacitors impregnated with the ether were manufactured by the Sprague Electric Company, North Adams, Massachusetts in an effort to develop radiation resistant electronic components. The capacitors were irradiated in the kilo-curie Cobalt-60 gamma facility of the Materials Central, Wright Air Development Division. Simultaneously, Sprague standard Vitamin "Q" mineral oil impregnated capacitors were irradiated for purposes of comparison.

WADD TR 60-645 (Continued)

Capacitance, dissipation factor and insulation resistance were measured throughout the tests lasting from 200 to 700 hours, depending upon the type of test employed. In most cases, the effects were an initial decrease and then an increase in capacitance, an increase in dissipation factor, and a large decrease in insulation resistance. The most dramatic effect was a marked decrease in capacitor lifetime in the gamma environment. Most failures were attributed to catastrophic breakup of the units due to internal gassing of the dielectric; it was this property which was significantly improved by the substitution of the ether for Vitamin "Q". In some instances, the BPPE units lasted three to four times as long as their Vitamin "Q" counterparts. However, the paper used in the capacitors was found to be a major contributor to the generated gases; therefore, the problem of catastrophic failure of the capacitors was only partially solved. In separate tests, both the Vitamin "Q" and the paper contributed enough gas individually to cause failure, while the DPPE essentially did not gas, thus, little was gained by the use of BPPE as the dielectric since the major culprit was the paper.

WADD TR 60-668, OTS Release

September 1960

SUBJECT: THE RADIATION CHEMISTRY OF CHLOROFORM SYSTEMS
INVESTIGATOR: Gabriel Stein, M. Ottolenghi, I. Eliezer
CONTRACT: AF 61(052)-224, Hebrew University, Jerusalem, Israel
ABSTRACT: A dosimeter based on solutions of biallyl in chloroform is developed. The radiation chemistry of pure H₂O and O₂ free chloroform is investigated and all products identified. A reaction mechanism is derived.

THERMO-PHYSICS

WADC TR 56-222, Part III, OTS Release

April 1960

SUBJECT: DETERMINATION OF EMISSIVITY AND REFLECTIVITY DATA ON AIRCRAFT STRUCTURAL MATERIALS - PART III. Techniques for Measurement of Total Normal Emissivity, Normal Spectral Emissivity at 0.665 Microns, Solar Absorptivity and Presentation of Results

INVESTIGATOR: O. Harry Olson, James C. Morris

CONTRACT: AF 33(616)-3002, Armour Research Foundation

ABSTRACT: Total normal emissivity is the ratio of total normal radiance emitted by a material to that emitted by an ideal blackbody at the same temperature. Parts I and II of this report described equipment and techniques employed for obtaining total normal emissivities of conducting materials, such as metals, at elevated temperatures. Part II describes equipment and techniques developed to make similar measurements on non-conducting materials such as refractories and glasses. Several samples are heated simultaneously in a tubular, wire wound furnace and presented successively in the field of view of a thermistor detector through a port in the furnace wall. The equipment and methods are described and data are presented for a list of materials. Low temperature measurements were made with equipment described in Parts I and II.

Normal spectral emissivity is the ratio of normal radiance emitted by a material to that emitted by an ideal blackbody at the same temperature at a given wave length. Most commonly, the wave length used is a narrow band at 0.665 microns and this is the sense in which the term is used in this report. Methods are described and results included.

An integrating sphere reflectometer was used to measure spectral reflectivity and transmission in the spectral range, 0.3 to 3.0 microns. This employs a Perkin-Elmer monochromator, a comparison integrating sphere, and a lead sulfide detector. Spectral reflectivity and transmission data are combined with solar spectral energy data to determine solar absorptivity at sea level and above the earth's atmosphere. Results are presented for a list of materials.

WADC TR 58-476

August 1960

SUBJECT: THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS VOLUME I. ELEMENTS (Melting Temperature Above 1000°F) REVISED EDITION

INVESTIGATOR: Alexander Goldsmith, Thomas E. Waterman, Harry J. Hirschhorn

CONTRACT: AF 33(616)-5212, Armour Research Foundation

ABSTRACT: Thermophysical property data, and their variation with temperature, are presented for a great number of solid materials, based on literature published during the period 1940-1957. Each reported value is shown and annotated, and recommended "most probable value" curves are given.

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Materials covered include Elements, Alloys, Ceramics, Cermets, Intermetallics, Polymerics, and Composite Materials. Except for materials in the last two categories, only those melting above 1000°F are included.

Properties covered include the following: Melting point, density, latent heats, specific heat, thermal conductivity, thermal diffusivity, emissivity, reflectivity, thermal expansion, vapor pressure, and electric resistivity.

Each of the four volumes is designed to be expandable, and it is expected that additional or revised data sheets for inclusion in these volumes will be forthcoming.

WADC TR 58-476, Volume II

November 1960

SUBJECT: THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS - VOLUME II.
ALLOYS (Melting Temperature above 1000°F)
INVESTIGATOR: Alexander Goldsmith, Harry J. Hirschhorn, Thomas E. Waterman
CONTRACT: AF 33(616)-5212, Armour Research Foundation
ABSTRACT: Thermophysical property data, and their variation with temperature, are presented for a great number of solid materials, based on literature published during the period 1940-1957. Each reported value is shown and annotated, and recommended "most probable value" curves are given.

Materials covered include: Elements, Alloys, Ceramics, Cermets, Intermetallics, Polymerics, and Composite Materials. Except for materials in the last two categories, only those melting above 1000°F are included.

Properties covered include the following: Melting point, density, latent heats, specific heat, thermal conductivity, thermal diffusivity, emissivity, reflectivity, thermal expansion, vapor pressure, and electric resistivity.

Each of the four volumes is designed to be expandable, and it is expected that additional or revised data sheets for inclusion in these volumes will be forthcoming.

WADC TN 59-210, OTS Release

April 1960

SUBJECT: EXPERIMENTAL INVESTIGATION OF THE DOWNSTREAM INFLUENCE OF STAGNATION POINT MASS TRANSFER
INVESTIGATOR: Paul A. Libby, Robert J. Cresci
CONTRACT: AF 33(616)-5944, Polytechnic Institute of Brooklyn
ABSTRACT: This report presents the results of an experimental investigation of the downstream influence of localized mass transfer in the stagnation

WADC TN 59-210 (Continued)

region of a blunt body under hypersonic flow conditions. The coolant is injected through a porous plug coaxial with the center line of symmetry of the model. The tests were carried out in a Mach 6 wind tunnel with stagnation temperatures of approximately 1600°R and with a stagnation pressure of approximately 600 psia. Four different gases were injected over a range of mass flows. The heat transfer on the impermeable section was measured under isothermal wall conditions; for the higher rates of mass flow, adiabatic surface temperatures were also determined. The theoretical analysis of the boundary layer flow is investigated in order to establish the similarity parameters for the flow system. These parameters permit the extrapolation of the test results to other flow conditions provided laminar flow prevails. Helium is found to be the most efficacious coolant.

WADC TR 59-366, Part I, OTS Release

May 1960

SUBJECT: THERMAL PROTECTION OF STRUCTURAL, PROPULSION AND TEMPERATURE SENSITIVE MATERIALS FOR HYPERSONIC AND SPACE FLIGHT - PART I. Relative Performance of Ablating Materials Exposed to Low and High Heat Flux Environments

INVESTIGATOR: John H. Bonin, Channon F. Price

CONTRACT: AF 33(616)-6006, Chicago Midway Laboratories

ABSTRACT: This report includes the results of an investigation for which the prime objective was the comparison of the relative performance of ablating materials exposed to thermal environments associated with recoverable space capsule trajectories. The experimental equipment used is briefly described, and test results for 19 different test specimens are presented. The data are used to compare the relative performance of the ablating materials with heat sink materials on a weight-per-unit-area basis.

WADD TN 60-32

April 1960

SUBJECT: AN APPARATUS FOR THERMAL ANALYSIS OF REACTIVE ALLOYS

INVESTIGATOR: Lt Donald J. Evans, Dr. Karl Strnat

ABSTRACT: An apparatus for thermal analysis of small alloy samples has been developed and tested. It is to be used in an experimental study of binary alloys containing a rare metal as one constituent. The alloys to be investigated are highly reactive. The apparatus is suitable for phase diagram work at temperatures up to 1500°C. It is almost fully automatic and yields a continuous record of the sample temperature vs time.

In this report, some general remarks on the method of thermal analysis are presented first, the apparatus is described, a few examples of heating and cooling curves obtained with the apparatus are shown, and the accuracy of the measurements is discussed on the basis of calibration runs.

WADD TN 60-88, OTS Release

June 1960

SUBJECT: ANALYSIS OF RELATIVE PERFORMANCE OF ABLATING MATERIALS
COMPARED TO A HEAT SINK MATERIAL

INVESTIGATOR: R. E. Otto

ABSTRACT: This report analyzes the data presented in WADC TR 59-366 generated by Chicago Midway Laboratories, University of Chicago, under Contract AF 33(616)-6006. Data are presented comparing relative performance of ablating materials with heat sink materials in terms of inside surface temperature rise and on a weight per unit area basis. Structural characteristics of available materials and analysis of re-entry deceleration loads are indicated.

WADD TR 60-463, Part I, OTS Release

October 1960

SUBJECT: THE VAPORIZATION AND PHYSICAL PROPERTIES OF CERTAIN
REFRACTORIES - PART I. Techniques and Preliminary Studies

INVESTIGATOR: A. A. Hasapis, M. B. Panish, C. Rosen

CONTRACT: AF 33(616)-6840, Avco Corporation

ABSTRACT: Simple effusion studies of the vaporization of rhodium, iridium, and osmium have been undertaken by the Knudsen effusion technique. The rate of effusion was determined by condensing the molecular beam on a target. Rhodium was studied from 2051 to 2205°K. In this temperature range, its heat of vaporization was found to be 129 Kcal/mole. Iridium was studied from 2100 to 2600°K. In this range, its tentative heat of vaporization is 155 ± 5 Kcal/mole. It was noted that some interaction between the iridium, the thoria liner, and the tungsten outer liner of the effusion cell may have occurred. No data were obtained for the vaporization of osmium because of extensive interaction between the thoria liner and the tungsten outer liner of the effusion cell.

Mass spectrometric studies have been made of the vaporization of thoria from tungsten effusion cells. Interactions between thoria and tungsten at elevated temperatures were noted. Tentative data on the vaporization of thoria to ThO and ThO₂ were obtained and are presented.

Mass spectrometric studies of the vaporization of Al₂O₃ in contact with tungsten have been made in conjunction with compatibility studies of tungsten and Al₂O₃. Data are given for the vaporization of Al₂O₃ in tungsten to yield Al, Al₂O, Al₂O₃, and WO₂. More information will be needed before thermodynamic calculations can be made for this system.

The continuously monitored effusion apparatus, the null-point effusion apparatus, the oscillating cup viscometer, and the sessile drop equipment have been constructed and calibrated.

The viscosity of fused silica has been determined at temperatures up to 2560°C where $\eta = 104.44$ poises. The viscosity of alumina has been measured using an iridium crucible up to 2200°C and determined to be approximately 13 poises.

SUBJECT: THERMAL PROPERTIES OF REFRACTORY MATERIALS
 INVESTIGATOR: Guy W. Lehman
 CONTRACTOR: AF 33(616)-6794, Atomics International
 ABSTRACT: A pulse heating method for measuring the specific heat of conductors from near absolute zero to their melting point is described. Results obtained on copper, iron, molybdenum, tantalum, and rhenium are reported here and indicate that the pulsed heating method gives specific heat data accurate to a few percent. Our measurements show that the heat capacities of molybdenum and tantalum gradually rise above their Dulong and Petit values of 0.06253 and 0.03316 cal/gm/ $^{\circ}$ C, respectively, at high temperatures. At 2800 $^{\circ}$ K, $cp(Mo) = 0.1350$ cal/gm/ $^{\circ}$ C and at 3200 $^{\circ}$ K, $cp(Ta) = 0.0667$ cal/gm/ $^{\circ}$ C. These anomalies have been noted in other transition metals.

The percentage elongation, relative to room temperature, of zirconium carbide has been measured between 1000 and 2000 $^{\circ}$ C and was found to vary linearly from 0.6% at 1000 $^{\circ}$ C to 1.6% at 2300 $^{\circ}$ C with a permanent set occurring above 2300 $^{\circ}$ C.

A transient method for measuring thermal diffusivity, specific heat, and thermal conductivity of insulators as well as conductors has been under development for the temperature range 20 $^{\circ}$ C to 2500 $^{\circ}$ C. A theoretical analysis of the transient thermal response of this system is presented and basic equations are derived for obtaining the above mentioned thermal properties. Theoretical and experimental results on the thermal response of a tantalum heater compare quite favorably. A cooling curve of beryllium oxide enclosed in a tantalum heater is presented and shows that heat conduction losses are completely negligible above 900 $^{\circ}$ C. Experimental diffusivity results are presented on Armco iron below the Curie point.

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE
 PART I. Mass Spectrometric Determination of the Dissociation Energies of the Molecules AgAu, AgCu, and AuCu
 INVESTIGATOR: Marcel Ackerman, Fred E. Stafford, J. Drowart
 CONTRACT: AF 61(052)-225, University of Brussels, Brussels, Belgium
 ABSTRACT: The vapors issuing from mullite and Graphite Knudsen cells containing pure metals and alloys of the triad Cu-Ag-Au have been analyzed mass spectrometrically. From the experimental ratios of diatomic to monoatomic species and the vapor pressures of the elements, the following dissociation energies are obtained.

$$\begin{array}{lll}
 D_{\infty}^0 (Cu_2) = 43.2 \pm 2.2 \text{ kcal} & D_{\infty}^0 = (AgAu) & 47.6 \pm 2.2 \\
 D_{\infty}^0 (Ag_2) = 35.7 \pm 2.2 \text{ kcal} & D_{\infty}^0 = (AgCu) & 40.7 \pm 2.2 \\
 D_{\infty}^0 (Au_2) = 51.9 \pm 2.2 \text{ kcal} & D_{\infty}^0 = (AuCu) & 54.3 \pm 2.2
 \end{array}$$

These are based on $H_{298}^{vap} = 81.1, 68.4$ and 87.5 cal/mole for Cu, Ag, and Au where

WADD TR 60-782, Part I (Continued)

D_o^0 of AgAu depends on the value for Cu, and D_o^0 of AgAu and AuCu on Au. The uncertainties quoted do not include the uncertainty in ΔH_{vap} .

The results are interpreted in terms of chemical bonding theories. The relation between bonding in the gas and in the pure condensed phases is again observed. Furthermore, a previously unobserved qualitative relation between the dissociation energy of the asymmetrical molecule and the heat of formation of the corresponding alloys is indicated.

ANALYTICAL TECHNIQUES

WADC TR 59-763

April 1960

SUBJECT: DEVELOPMENT OF AN INFRARED, PRISM-GRATING, DOUBLE BEAM RECORDING SPECTROPHOTOMETER
INVESTIGATOR: Hamilton W. Marshall
CONTRACT: AF 33(616)-5190, Perkin-Elmer Corporation
ABSTRACT: The design and construction of a Double Beam Recording Wide Range Infrared Spectrophotometer to automatically cover the range from 2.5 to approximately 50 microns through four ranges is discussed. Experiments with gratings showed that to cover this range, gratings blazed at 6 microns through 30 microns, both used in the first and second orders were required. By selecting the beginning and end points of the regions as all exact integral multiples of one another, one wave number cam is used to drive the gratings thus assuring simple and accurate abscissa presentation. In order to separate the overlapping orders of the gratings, three prisms are used, NaCl, KBr, and CsI, with the prism monochromator double passed, since the dispersion from the prism must be large when wide slits are used to gain high signal-to-noise ratios. Because slit widths can become as great as 20 mm, an attenuator using two blades which may be tilted, similar to the action of a venetian blind, is used. A Golay cell was chosen as the detector since a large target was necessary to receive the energy from the wide slits.

WADD TR 60-204, OTS Release

September 1960

SUBJECT: INVESTIGATION OF THE INFRARED ABSORPTION OF SELECTED AROMATIC COMPOUNDS IN THE REGION OF 2 TO 35 MICRONS
INVESTIGATOR: R. J. Jakobsen
CONTRACT: AF 33(616)-6140, Battelle Memorial Institute
ABSTRACT: Complete vibrational assignments have been made for phenol, p-xylene, p-cresol, and hydroquinone. All assignments of ring vibrations have been checked by use of the Pitzer-Scott modification of the Teller-Redlich product rule. These complete vibrational assignments have been made in order to provide a foundation for the assignment of the low-frequency vibrations of a series of para-substituted phenols.

Infrared spectra of the selected para-substituted phenols have been recorded over the 2- to 35-micron range. With the aid of these infrared data and of Raman spectra, most of the low-frequency vibrations have been assigned. The reliability of the assignments and the significance of the assignments are discussed, as well as the determination of the modes of vibration.

Correlation studies of frequency with physical properties have been made for the assigned frequencies that are sensitive to the nature of the substituent. Vibration 20' still shows (although not as good as shown in Progress Report No. 1) a correlation with the substituent constants, σ_p and σ_R , and possibly shows a correlation with Platt's spectroscopic moments. The

WADD TR 60-204 (Continued)

possible correlations of $\nu_{18'}$ with group mass and $\nu_{20'}$ with Sanderson's partial charges are discussed. These last two correlations may indicate the need for multiple correlations since, for instance, $\nu_{18'}$ seems to give one correlation with group mass of certain types of phenols, and another correlation with group mass for other types of phenols (depending on the type of substituents).

WADD TR 60-333, OTS Release

September 1960

SUBJECT: FAR INFRARED SPECTRA OF SUBSTITUTED AROMATIC HYDROCARBONS
INVESTIGATOR: William S. Wilcox, C. V. Stephenson, W. C. Coburn, Jr.
CONTRACT: AF 33(616)-5855, Southern Research Institute
ABSTRACT: The results of a fundamental study of the infrared spectra of substituted aromatic hydrocarbons from 2 to 35 microns are discussed. The purpose of this work has been to explore the possibility of correlating the absorption frequencies of substituted benzenes with physical properties of the molecules or of their substituents. Since the far infrared spectra (15 to 35 microns) exhibits a greater sensitivity to changes in the substituents bonded to the aromatic ring than the rocksalt spectra (2 to 15 microns), the correlation studies have been limited to the long wavelength region. The materials apparatus and techniques used in this study are discussed in Appendix I. The far infrared spectra of 94 substituted benzenes are given.

WADD TR 60-415, OTS Release

September 1960

SUBJECT: ELECTROANALYTICAL METHOD FOR THE DETERMINATION OF CARBON AND HYDROGEN IN ORGANIC COMPOUNDS
INVESTIGATOR: Herbert S. Haber, Kenneth W. Gardiner
CONTRACT: AF 33(616)-6676, Bell & Howell Research Center
ABSTRACT: This report contains the results of research investigations completed for Contract AF 33(616)-6676 during the period from June 15, 1959 to May 14, 1960. Research efforts were directed toward the development of a rapid electroanalytical method for the determination of carbon and hydrogen in non-metallic organic compounds containing carbon, hydrogen and oxygen. The method utilizes a novel combustion technique and is applicable to the solid, liquid or gaseous state of aggregation. The method at the present time is specific for carbon and hydrogen only in the presence of oxygen, but future work is planned to extend the applicability to compounds containing halides, sulfur, nitrogen, etc. The time required for analysis of a sample is approximately 10 minutes including the weighing of the sample. A suitable-packaged prototype instrument designed to accomplish these ends will be available for delivery to WADD at the termination of this contract.

DESIGN CRITERIA

WADC TR 55-319, Sup 2

September 1960

SUBJECT: EFFECTS OF WEATHERING ON THE MECHANICAL PROPERTIES OF FOUR REINFORCED PLASTIC LAMINATES
INVESTIGATOR: Kenneth Kimball
CONTRACT: DO 33(616)-58-1, Forest Product Laboratory
ABSTRACT: Four different reinforced plastic laminates were subjected to outdoor weathering at three sites having entirely different weather conditions. After completion of the exposure, the laminated panels were tested in flexure at the U. S. Forest Products Laboratory. Data on the effect of 3 and 12 months' exposure on the flexural properties and the appearance of the laminates are presented in this report.

Another series of panels are still undergoing exposure at all three sites, and this series will be tested after 3 years' exposure. Upon completion of the tests on these panels, all data will be compiled in a final report.

Data obtained from the tests to date show that the effect of outdoor exposure varies greatly with different types of laminates and conditions of exposure. The greatest loss in strength was usually associated with exposure at the Florida site. In general, normal and wet flexural strengths of weathered glass-fabric laminates were 90 percent or more of the control strengths, but the high-temperature strength was only about 60 to 80 percent of the control. The phenolic-asbestos laminate had a higher over-all strength retention than the glass-fabric laminates.

Results of strength tests of over 700 specimens are presented in this interim report.

WADC TR 57-344, Part IV, OTS Release

May 1960

SUBJECT: DEVELOPMENT OF NIOBIUM-BASE ALLOYS
INVESTIGATOR: Richard T. Begley, William N. Platte
CONTRACT: AF 33(616)-5754, Westinghouse Electric Corporation
ABSTRACT: Vacuum tensile data were obtained for pure niobium from room temperature to 1371°C (2500°F). The data for niobium followed the general pattern exhibited by other pure refractory metals. The ductile-brittle transition (in impact) for commercial purity, arc melted niobium was close to room temperature.

The effect of binary additions of Ti, Zr, Hf, V, Mo, W, Re, Al, and Y on the hardness and workability of niobium was determined. Nb-Ti and Nb-Y alloys had excellent cold workability.

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Mechanical property data were obtained at room temperature and 1093°C (2000°F) on niobium containing additions of Ti, Zr, Hf, V, Mo, and W. Of the elements studied, vanadium additions were the most effective strengtheners. Nb-Zr and Nb-Hf alloys having high oxygen contents had very good high temperature properties. It appears that the interaction of oxygen with the alloy addition may be responsible for the high strength. Tensile data were also obtained on alloys having ternary and quaternary additions of Ti, Zr, Hf, and Mo. Nb-Ti-Zr-Hf alloys exhibited yield strengths well in excess of 40,000 psi at 1093°C (2000°F).

Welding studies on a number of niobium alloys containing additions of Ti, Zr, Hf, V, and W were carried out. Satisfactory welds were obtained in all the alloys studied. Bend test data were obtained on the weld specimens.

WADC TR 57-374, Part V, OTS Release

October 1960

SUBJECT: PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS - PART V.
Thermal Diffusivity of Magnesia-Stabilized Zirconium Oxide
at High Temperatures

INVESTIGATOR: Howard W. Flieger, Jr., Friedrich P. Knudsen, Defoe C.
Ginnings

CONTRACT: AF 33(616)-56-21, National Bureau of Standards

ABSTRACT: As a part of a program developing standards of thermal conductivity and thermal diffusivity of refractory materials at high temperatures, thermal diffusivity measurements have been made on dense polycrystalline zirconium oxide which had been stabilized with magnesium oxide. The results indicate that the stabilization of the zirconium oxide was inadequate, giving progressively increasing values of thermal diffusivity after cycling to temperatures of about 1200°C. Thermal expansion and X-ray measurements indicate that the increase in thermal diffusivity values correspond to a change from cubic to monoclinic crystalline form.

WADC TR 59-744, Volume V, OTS Release

November 1960

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT
RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS
VOLUME V. Mechanical Properties of Bare and Coated
Molybdenum Alloy

INVESTIGATOR: J. Robert Kattus, James B. Preston, Herman L. Lessley

CONTRACT: AF 33(616)-6034, Southern Research Institute

ABSTRACT: The mechanical properties of arc-cast, fully recrystallized molybdenum 1/2%-titanium alloy - bare, W-2 coated, and Durak-MG coated -- were determined at temperatures up to 3000°F in short-time tension and compression and in tension and compression creep for times up to 10 hr. The purpose of the work is to provide data, for this alloy upon which to base the design of leading edges for hypersonic boost-glide vehicles.

WADC TR 59-744, Volume V (Continued)

In inert atmospheres, this molybdenum alloy retains useful strength and good ductility at temperatures up to 3000°F. Both coatings have an extreme embrittling effect and a weakening effect on the base metal at room temperature. At 1200°F and above, the coated material is equivalent in strength and only slightly less ductile than the bare material. When properly applied, both coatings provide good oxidation protection in still air at temperatures up to 3000°F and for times up to 10 hr. Under tension and compression loads, the coatings can absorb, without failure, elastic strain and varying degrees of plastic strain from less than 1% up to 18% depending upon the conditions.

With W-2 or Durak-MG coatings of optimum quality, the molybdenum 1/2%-titanium alloy is recommended for applications that are limited to no plastic strain at room temperature and to small amounts of plastic strain at elevated temperatures to a maximum of 3000°F for times up to 10 hr. Although the coatings can absorb considerable plastic strain under many conditions, further research is needed to safely extend their usefulness beyond small amounts of plastic strain at 2100°F and at room temperature. Future research is recommended to minimize or to eliminate the embrittling effects of coatings on the base material and to develop quality-control methods to assure consistent optimum quality in the protective coatings.

WADC TR 59-744, Volume VI, OTS Release

November 1960

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME VI. Determination and Design Application of Mechanical Properties of Bare and Coated Graphite

INVESTIGATOR: Alfred Rudnick, Robert L. Carlson, George K. Manning

CONTRACT: AF 33(616)-6034, Battelle Memorial Institute

ABSTRACT: The purpose of this contract was to investigate the feasibility of utilizing available heat-resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This volume presents the results of a study on the mechanical properties of a siliconized Grade ATJ graphite. The mechanical properties were studied as functions of temperature (to 2950°F), strain rate, size, and stress state. Supplementary studies were conducted to determine the room temperature properties of the uncoated material and the degree of anisotropy of both the coated and uncoated material.

Cross variations in the material tended to obscure the effects of the parameters under consideration. Inspection techniques were developed, however, which will reduce these variations. With proper selection of material quality, it is believed that the siliconized Grade ATJ graphite can be used effectively as a structural component at elevated temperature in an oxidizing atmosphere.

SUBJECT: INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME VII. Oxidation Resistance of Bare and Coated Molybdenum Alloy and Graphite

INVESTIGATOR: Donald J. Powers, John A. Dickson, Joseph C. Conti, Frank M. Anthony

CONTRACT: AF 33(616)-6034, Bell Aerosystems Company

ABSTRACT: The purpose of this contract was to investigate the feasibility of utilizing available heat resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This particular volume presents the results of oxidation resistance tests of bare and coated 0.5% titanium alloy of molybdenum and ATJ graphite. Chromalloy W-2 and Durak-MG coatings were evaluated on the molybdenum alloy while the ATJ graphite was coated by the National Carbon Company's siliconizing process.

Tests were conducted in five different facilities including three arc plasma jets. Specimen temperatures ranged from 2000°F to 3400°F. Test times ranged from approximately 1 minute to 4 hours.

Both uncoated materials reacted exothermically under oxidizing conditions. Surface recession was the major result of the high temperature exposure in the oxidizing environments. The surface recession was quite predictable and uniform; it was a function of specimen temperature and environmental pressure.

A high degree of failures were encountered on all coated specimens. The failures were dependent on time and temperature but there was considerable scatter in the results. For the coated molybdenum specimens the edges and corners were most prone to failure. Failures of the siliconized ATJ specimens were more or less randomly located over the surface area.

SUBJECT: A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE (PHASE I) PART I. Properties of Fluids

INVESTIGATOR: Victor J. Johnson, General Editor

CONTRACT: AF 33(616)-58-4, National Bureau of Standards

ABSTRACT: This first phase of the Compendium covers ten properties of ten fluids (Part I), three properties of solids (Part II), and an extensive bibliography of references (Part III). Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, adsorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

WADD TR 60-56, Part II, OTS Release

October 1960

SUBJECT: A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE (PHASE I) PART II. Properties of Solids

INVESTIGATOR: Victor J. Johnson

CONTRACT: AF 33(616)-58-4, National Bureau of Standards

ABSTRACT: This first phase of the Compendium covers ten properties of ten fluids (Part I), three properties of solids (Part II), and an extensive bibliography of references (Part III). Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, adsorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

WADD TR 60-56, Part III, OTS Release

October 1960

SUBJECT: A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE (PHASE I) PART III. Bibliography of References (Cross-Indexed)

INVESTIGATOR: Victor J. Johnson, General Editor

CONTRACT: AF 33(616)-58-4, National Bureau of Standards

ABSTRACT: This first phase of the Compendium covers ten properties of ten fluids (Part I), three properties of solids (Part II), and an extensive bibliography of references (Part III). Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, adsorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

WADD TR 60-363, OTS Release

November 1960

SUBJECT: INVESTIGATION INTO "MORE COMPLETE USE OF STRUCTURAL MATERIALS" THROUGH A STUDY OF THE STRESS-TEMPERATURE-TIME CONDITIONS OF A RE-ENTRY VEHICLE

INVESTIGATOR: J. E. Davis, J. E. Fischler, J. W. Lobbett, D. Saltman, J. W. Stanwood, N. A. Tiner

CONTRACT: AF 33(616)-6680, Douglas Aircraft Company, Inc

WADD TR 60-363 (Continued)

ABSTRACT: In the past, vehicles operated in a fairly low temperature environment. The selection of materials were primarily dependent on the quasi-static and dynamic loads. With the advent of manned hypersonic glide re-entry vehicles, the temperature and time parameters were found to significantly affect the selection of materials. In this report, the criteria for including the effects of temperature and time are clarified by using six "measuring sticks". Two materials, Inconel 718 and Haynes 25 were evaluated for repeated exposure to a typical hypersonic re-entry glide trajectory.

WADD TR 60-404, OTS Release

January 1961

SUBJECT: A STUDY OF THE EFFECT OF ELECTRON BEAM MELTING ON COMPOUNDS AND METALS

INVESTIGATOR: R. L. Martin, S. R. Seagle, O. Berteau

CONTRACT: AF 33(616)-5603, Reactive Metals, Inc

ABSTRACT: Because of the high temperatures and vacuum that can be obtained during electron beam melting, this process offers the potential of purifying high melting point materials. This program consists of a study of the effect of the electron beam melting process on boron, boron-carbon alloys, tantalum carbide, titanium carbide, zirconium di-boride, hafnium, tungsten, cobalt, vanadium and beryllium. In addition, deoxidation studies were carried out on beryllium, vanadium, cobalt and molybdenum. When possible, microstructures, tensile properties, and chemical compositions of the materials melted were obtained.

The compounds studied either could not be melted or were brittle after melting. The interstitial content of all the metals was reduced by the electron beam melting process. By the addition of deoxidants, the interstitial contents of molybdenum and beryllium were reduced. Improvement in the tensile properties of vanadium and molybdenum were obtained by the addition of deoxidants to these metals.

WADD TR 60-419, OTS Release

October 1960

SUBJECT: EFFECTIVE STRESS CONCENTRATION FACTORS FOR FLIGHT VEHICLE MATERIALS UNDER VARIOUS CONDITIONS DURING FATIGUE TESTING

INVESTIGATOR: V. F. Lardenoit

ABSTRACT: This report presents in the form of tables and curves, the effective stress concentration factors, K_f , for a number of aircraft materials subjected to various conditions during fatigue testing. The influence on K_f is shown for such parameters as temperature, stress ratio, specimen size, direction of testing, test frequency, fabrication of notches, heat treatment, grain size, and for titanium and its alloys, interstitial constituents and hydrogen level.

Because K_f is sensitive to so many variables, no attempt is made to relate it to any of the above listed parameters.

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WADD TR 60-489, OTS Release

October 1960

SUBJECT: INVESTIGATION TO DEVELOP OPTIMUM PROPERTIES IN FORGED
Ti-7Al-4Mo
INVESTIGATOR: James E. Hamer
CONTRACT: AF 33(616)-6122, Crucible Steel Company of America
ABSTRACT: The influence of six processing sequences on the micro-structure, mechanical properties and heat treatment response of the Ti-7Al-4Mo alloy has been studied. The information developed demonstrates the critical effects of processing history in controlling the material performance levels and emphasizes the importance of such considerations in attaining "optimum" values.

Forging entirely above the beta transus produced the usual Widmanstatten type of structure found in titanium alloys after such processing. As expected, the creep resistance of this structure was excellent; however, heat treatment response and tensile ductility were poor, and a reduction in charpy impact strength of 3/4" rod was noted for all thermal treatment conditions. Forging entirely below the beta transus developed an equiaxed micro-structure and generally good properties except for reduced creep resistance. Duplex forging, i.e., forging initially above the beta transus, cooling to room temperature, and reheating to below the beta transus for finish forging, produced a nearly equiaxed microstructure and general property levels quite similar to all below-betaforging but with improved creep resistance.

In general, notch tensile and notch stress rupture did not show any direct relationships with processing history. Notch rotating beam fatigue tests for above-beta forged material showed more scatter than those on material forged below the beta transus though the endurance limit was not greatly affected.

WADD TR 60-793, OTS Release

March 1960

SUBJECT: DIFFUSION IN REFRACTORY METALS
INVESTIGATOR: N. L. Peterson
CONTRACT: AF 33(616)-7382, Advanced Metals Research Corporation
ABSTRACT: Data available in the open literature on diffusion in tungsten, tantalum, molybdenum, niobium, platinum, hafnium, zirconium, vanadium, chromium, and titanium is reviewed and evaluated. Information on ninety-five binary systems and thirteen ternary or higher order systems is reported.

Care was taken to make this report as complete as possible at this time. For some systems where no diffusion data was available, diffusion coefficients were estimated from existing theories or from data on diffusion controlled processes.

APPLICATION STUDIES

WADC TR 57-649, Part III, OTS Release

November 1960

SUBJECT: DETERMINATION OF THE MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING - PART III. The Effect of Simultaneous Heating and Loading on the Tensile Properties of Typical Structural Alloys

INVESTIGATOR: A. Clyde Willhelm, J. Robert Kattus

CONTRACT: AF 33(616)-3494, Southern Research Institute

ABSTRACT: Three series of experiments were conducted to determine the effects of simultaneous heating and loading on the stress-strain characteristics of some typical structural sheet metals, and to correlate the experimental data with predicted effects based on the concept of a Mechanical Equation of State. The first series of experiments was performed on Inconel X nickel-chromium age-hardening alloy and on 17-7 PH/TH 1050 stainless steel. The 17-7 PH and full-hard Type 301 stainless steel were used for the second and third series respectively. Improvements in the experimental method and equipment were sought in each succeeding series.

The results of the experiments indicate that although practical application of the concept of a Mechanical Equation of State to design problems appears promising on a qualitative basis, improvements in prediction and experimental methods will be required to obtain quantitative confirmation.

WADC TR 59-366, Part II, OTS Release

July 1960

SUBJECT: THERMAL PROTECTION OF STRUCTURAL, PROPULSION, AND TEMPERATURE-SENSITIVE MATERIALS FOR HYPERSONIC AND SPACE FLIGHT

INVESTIGATOR: William P. Manos, Donald E. Taylor, Anthony J. Tuzzolino

CONTRACT: AF 33(616)-6006, Chicago Midway Laboratories

ABSTRACT: This report presents the results of an investigation intended to examine various phenomena applicable to the protection of materials subjected to severe thermal environments and evaluate their effectiveness on the survival of materials now available. The heating experienced by materials in selected severe thermal environments is reviewed. The various phenomena that might be employed for protection are studied and evaluated analytically. Experimental tests were performed to aid in evaluation of certain phenomena, using the plasma discharge of a 1000-KW air-stabilized arc to provide a severe thermal environment. These results are reported, along with a correlation of the observed behavior with theory and previous tests.

WADD TR 60-58

May 1960

SUBJECT: SYMPOSIUM ON PROCESSING MATERIALS FOR RE-ENTRY STRUCTURES
INVESTIGATOR: Materials Central

WADC TR 53-373, Sup 8

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WADD TR 60-58 (Continued)

ABSTRACT: The theme of the symposium, "Processing" is carried throughout the papers which cover general requirements and problem areas, materials applications, methods used to form and fabricate items from high strength moderate temperature materials, high temperature metallics and high temperature non-metallics. Specific papers concern themselves with methods for processing materials such as: refractory ceramics, graphite and ablation materials, refractory metals, super alloys, forging methods, explosive forming, welding and plasma and metallizing. There is a limited amount of published information for most of these topics.

WADD TN 60-246, OTS Release

February 1961

SUBJECT: MATERIALS INFORMATION CENTERS
INVESTIGATOR: K. A. Winter, Irving Lopatin
CONTRACT: AF 33(616)-6288, McGraw-Hill Book Company, Inc
ABSTRACT: This technical note contains a listing of all known organizations or agencies which may be designated as a materials information center on the basis of their activities concerned with the collection and dissemination, in some manner, of information on a class, group, or type of material, or materials.

The purpose of this technical note is to provide interested personnel of the Department of Defense, its contractors, and materials suppliers with a means of locating and obtaining information pertinent to their specific area of interest.

WADD TR 60-584, OTS Release

October 1960

SUBJECT: HANDBOOK OF FIBROUS MATERIALS
INVESTIGATOR: Harry Mileaf
CONTRACT: AF 33(616)-5726, McGraw-Hill Book Company
ABSTRACT: This report contains the summarized and consolidated information extracted from WADD Technical Reports covering several phases of fibrous materials research. The information is arranged to make the results of these reports more readily available and useful to decelerator designers and others interested in the fibrous materials phase of Air Force research.

This report is divided into sections covering the pertinent facets of fibrous material information. Fairly complete information is supplied on various phases of basic design data, sewability, friction, abrasion and weather resistance, porosity and air permeability, temperature properties and chemical resistance of different yarns, cords, webbings and fabrics. Some information is also supplied on sunlight and weather resistance, tear resistance, radiation, aging and electrostatic properties, stiffness, flexibility, elongation, elastic recovery and energy absorption.

February 1961

SUBJECT: CONSIDERATIONS AND RECOMMENDATIONS FOR DEVELOPING A
MATERIALS INFORMATION PROCESSING CAPABILITY

INVESTIGATOR: Albert J. Belfour

CONTRACT: AF 33(616)-7064, Belfour Engineering Company

ABSTRACT: A survey is summarized in conjunction with recommendations for the development of a Materials Information Processing Capability at the WADD Materials Central. The discussion covers some basic concepts of technical information, user requirements, and existing information "centers". A consistency in pattern of information processing techniques is examined and discussed. Recommendations are given for the development of a Materials Information Processing Capability.

ACOUSTICAL MATERIALS AND STUDIES

WADC TR 59-676, OTS Release

March 1961

SUBJECT: WADC - UNIVERSITY OF MINNESOTA CONFERENCE ON ACOUSTICAL FATIGUE

INVESTIGATOR: W. J. Trapp, D. M. Forney, Jr.

ABSTRACT: This report is composed largely of invited papers and some selected seminar material presented at the Conference on Acoustical Fatigue held at Beecher's Resort, Annandale, Minnesota, from 29 September 1959 through 2 October 1959. The conference was organized jointly by the Materials Laboratory of Wright Air Development Center and the University of Minnesota. It was designed to establish communication between individuals from the several disciplines involved in acoustical fatigue problems, namely, acoustics, applied mechanics, and service failure analysis. It was hoped thereby that an improvement in the over-all understanding of the acoustical fatigue problem would ensue. Another important objective was to define clearly the critical problem areas and aspects which would require emphasis in the future. Over 100 participants from the United States and abroad were invited to attend and take part in the discussions. A list of attendees is included in this report.

WADD TR 61-70, OTS Release

March 1961

SUBJECT: EXPERIMENTAL STUDY OF THE RANDOM VIBRATIONS OF AN AIR-CRAFT STRUCTURE EXCITED BY JET NOISE

INVESTIGATOR: B. L. Clarkson, R. D. Ford

CONTRACT: AF 61(052)-332, University of Southampton, United Kingdom

ABSTRACT: Recordings have been made of the strains induced in a full scale rear fuselage test structure of the Caravelle airliner when one jet engine is running at maximum take-off thrust. The structure is a conventional sheet-stringer combination attached to pressed out frames.

The analysis has been concentrated on the strains in the centres of panels. Correlation measurements have indicated that the lower frequencies (up to 500 c.p.s.) are associated with overall vibration modes and have low strain amplitude. The larger panel strains occur at higher frequencies with the frames acting as boundaries. In these measurements the main resonance peak in each panel occurs at about 600-700 c.p.s. and has been identified with the fundamental stringer twisting mode (i.e., adjacent panels 180° out of phase). There are generally two smaller peaks in the 800-1000 c.p.s. range but the modes of vibration have not been completely identified due to lack of information.

An attempt has been made to calculate the panel resonant frequencies theoretically, assuming that the frames act as boundaries. Although this work appears promising it has not yet progressed far enough for any definite conclusions to be drawn from it.

WADC TR 53-373, Sup 8

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ELECTRONICS

AMC TR 60-7-352

1 November 1960

SUBJECT: IN-LINE EXHAUST MACHINE
INVESTIGATOR: H. K. Trinkle
CONTRACT: AF33(600)-26222, General Electric Company
ABSTRACT: This report describes functional improvements made in the Receiving Tube Inline Exhaust Machine and its application in process experiments performed, using tubes type 6005. The work program was based on the experience gained during work on tubes type 5654, wherein three major areas of difficulty were encountered.

One source of trouble was due to the recurrent malfunction of various machine components. Improvements have been incorporated on the machine which have resulted in a very real improvement in operation. The performance of the machine during 6005 process experiments has been quite satisfactory.

Another source of difficulty encountered in the earlier work was attributed to the choice of the 5654 as the first test vehicle. The 5654 is a relatively low power dissipation type and its quality has been carefully optimized by an extended period of development and production. On the other hand, the 6005 is a high power dissipation tube and has a history of life impairment due to the evolution of gas. The 6005 should therefore comprise a more satisfactory test vehicle to demonstrate the advantages of the inline exhaust process. Life tests on a limited number of tubes have indicated an improvement in performance due to the process, but the results are still not as impressive as had been expected.

The final important question that was encountered in the previous work was taken from a very approximate analysis of the manifold pressure record during processing. The analysis indicated that the residual pressure level in the tube at the end of the process might be much higher than had been expected. Special equipment has been constructed and used in process experiments on the 6005 to provide more accurate information. These experiments have substantiated the conclusion taken from the 5654 manifold pressure data. The experiments indicate that the outgassing characteristics of the 6005 impose a basic limitation on the residual gas level that may be attained by any practical process schedule. The degree of outgassing attainable within the present operating limits of the machine is controlled primarily by the complex desorption characteristics of the plate and cathode, acting in combination with pumping restrictions due to the tubulation and micas. Variations in process parameter levels over a broad range seem to have very little effect on the total amount of gas evolved.

A description of process experiments and life tests on subminiature tubes type 5902 is included in the report.

AMC TR 60-7-444

October 1960

SUBJECT: DEVELOPMENT OF THE WAFER TRANSFORMER
INVESTIGATOR: Colonel A. Hughes
CONTRACT: AF33(600)-30839, American Machine & Foundry Co.
ABSTRACT: The primary purpose of this program was to establish production techniques and to "design-fabricate" semi-automatic equipment for the production of wafer transformers. The wafer transformer coil is wound with a metallic foil ribbon instead of the conventional round wire. The flat wafer or pancake form of the winding used, permits very compact construction and results in the reduction of transformer weight and size by up to 50%. Electrical and environmental testing to Specification MIL-T-27A has shown these transformers to be reliable and satisfactory in performance. Approximately 275 transformers have been fabricated and tested. These designs have included both aluminum and copper ribbons, classes R and U and grades 4 and 5 of Specification MIL-T-27A, and ribbon sizes from AWG 10 to 37. A full study of the method of insulating a ribbon, and of the characteristics of both soldered and welded aluminum-copper junction has been completed. Experimental pilot line equipment for producing ribbon conductor (both insulated and not insulated), for winding wafer coils, for interconnection of wafers, and for the assembly of coil and core, has been developed, and is described in Volume II of this report.

The Air Force, faced with an exponential rise in military aircraft electronic content, is sponsoring this program so that the above noted benefits of size, weight and production may be realized.

AMC TR 60-7-505

May 1960

SUBJECT: DEVELOPMENT OF PRODUCTION PROCESSES & TECHNIQUES FOR HIGH VOLTAGE SILICON RECTIFIERS
INVESTIGATOR: Ray Kuhl
CONTRACT: AF33(600)-36110, Texas Instruments Incorporated
ABSTRACT: High-voltage silicon rectifiers in the 20,000; 30,000; and 60,000 P.I.V. range for use as clipper diodes in ground and airborne electronic applications can be economically fabricated in production quantities.

Successful operation of the rectifiers at extremely high voltages was made possible by resolution of the problems associated with avalanche breakdown, junction forming by diffusion, arcing, stacking of basic cells and packaging. Solution to the problem of arcing under evacuated conditions and packaging contributed most to the success of the program.

Test samples furnished to a contractor for application in a fire control system were satisfactory under all conditions.

AMC TR 60-7-534

March 1961

SUBJECT: DRY CIRCUIT TESTS & TEST EQUIPMENT FOR ACCEPTANCE
TESTING OF RELAYS FOR LOW LEVEL APPLICATION

INVESTIGATOR: H. K. Trinkle

CONTRACT: AF33(600)-33402, Union Switch & Signal Co.

ABSTRACT: The general performance and reliability of a relay with respect to surety of contact make appears to depend upon two main factors: design and manufacturing processes.

Particulate matter apparently causes the major portion of contact failures. That is, the number of failures for a given period of operation depends upon the amount of contamination in the relay and its random movement.

Design can reduce or eliminate the problem due to particle contamination by insuring the high contact force and the relatively large amount of contact wipe required to maintain clean contact interfaces. Proper material selection for the moving parts will help to maintain wear product contamination at a minimum value.

Manufacturing processes can help produce a reliable product by reducing to a minimum the contamination included in each unit during its construction.

It is important that those tests which attempt to determine contact reliability maintain operation in the low level area for each total contact operation to obtain the most accurate results; thus, the electrical test parameters must be as low as possible. It is also obvious that in attempting to measure a factor which indicates product quality or reliability, the test equipment must have high reliability.

Four different makes of relays, thought to be representative for hermistically sealed relays, were selected for this study and test.

AMC TR 60-7-535

May 1960

SUBJECT: ELECTRON TUBE VIBRATION TEST PROCEDURES

INVESTIGATOR: W. F. Gilmer

CONTRACT: AF33(600)-31879, Armour Research Foundation

ABSTRACT: An intensive investigation was made of vibration test procedures applied to tube types 5654/6AK5W/6096, 5670WA, 5718, 5840, 6101/6J6WA, 6112 and 6189/12AU7WA. Effort on the program was divided into three tasks.

Task A was concerned with fatigue tests of tube types 5654/6AK5W/6096, 6101/6J6WA and 6189/12AU7WA. Correlation studies were made of fatigue effects produced by the 96-hour fatigue test specified in Par. 4.9.20.6 of MIL-E-1C, and the 3-hour fatigue test

AMC TR 60-7-535 (Continued)

specified in Par. 4.7.1.2 of MIL-E-5272A. The extent of tube degradation was evaluated in terms of measurement of tube parameters and measurement of tube noise during subjection to a "standard" vibration measurements test. Heater to cathode leakage was found to be the most sensitive indicator of tube degradation. Short duration, increased severity fatigue tests were also investigated.

Task B considered evaluation of a vibration measurements test for the purpose of establishing a standard test. Tube types 5654/6AK5W/6096, 5670WA, 5718, and 5840 and 6112 were studies. Three vibration tests were compared, two at fixed frequency, fixed acceleration level. Maximum tube noise was the data of the test. Design of experiments considered and evaluated variations in noise voltage due to: differences between tube types; differences between manufacturers for each tube type; differences between lots for each manufacturer; differences between tubes within a given lot; random variations between readings on the same tube due to measurement instruments and/or recording equipment characteristics; and variations between readings on the same tube due to carryover effects from its use history. Test reproducibility for fixed frequency test conditions lead to the conclusion that only limited correlation could be obtained from different tests or, in fact, repetition of the same test.

In task C, factors affecting tube noise were studies in detail. Swept sinusoidal tests and random excitation tests (constant spectral density and equal energy per octave) were studied. Effects of acceleration level, sweep rate, bandwidth were studies with respect to tube noise magnitude and harmonic content, over a wide dynamic range. Major tube resonances were determined, both for principle frequencies at which they occurred and the Q's of resonance. Reproducibility of test results were studies where the same vibration test was performed on a given tube using two different vibration excitors.

AMC TR 60-7-608

June 1960

SUBJECT: MINIATURIZED TERMINALS FOR HIGH-VOLTAGE, HIGH-
ALTITUDE TRANSFORMERS
INVESTIGATOR: W. F. Gilmer
CONTRACT: AF33(600)-37392, General Ceramics Div., Indiana
General Corp.
ABSTRACT: A general description of the process employed by Advanced Vacuum Products is given, and the changes in the original terminal designs (as set forth in WADC Technical Report 56-658) that were necessitated by the adoption of the Advac process are described.

Twelve terminals of each of the three types (total of 36) were fabricated and then tested in accordance with the

AMC TR 60-7-608(Continued)

qualification requirements of Exhibit WCRE 57-2A. The terminals successfully passed all the qualification tests except the contact resistance specification. A slight change in manufacturing procedure was recommended in order to meet the contact resistance specification.

For all three types of terminals, the procedure for attaching the proper cable to the terminal is described in detail.

350 of each type of terminal (total 1050) were fabricated and delivered to the Government. During production of these terminals inspection tests devised by the contractor and approved by the contracting officer were performed. The terminals met the acceptance levels agreed upon by the contractor and the Government.

The process of manufacture is described in detail, including processing data. The requirements with respect to personnel, equipment and floor space are set forth, and a recommended equipment layout for the manufacture of 375 terminals per day (125 for each of the three types) is presented.

AMC TR 60-7-663

June 1960

SUBJECT: HIGH POWER SILICON TRANSISTORS
INVESTIGATOR: Ray Kuhl
CONTRACT: AF33(600)-36234, Westinghouse Electric Corp.
ABSTRACT: It has been demonstrated that the Westinghouse alloyed silicon transistor meets the objectives required. The transistor has proven to be a manufacturable design which ensures commercial availability. Two devices were produced with collector current ratings of 5 and 15 amperes, the first device being completed in Jan 1959. Since then, the 5 ampere device has been in full-scale production and is now commercially available in quantities. The second device is now designated as Westinghouse Type WX115B, and has collector current rating of 15 amperes and collector breakdown voltage of 120 volts. The final design has an operating voltage of 100 volts which is several times that of the contract objective of 30 volts.

AMC TR 60-7-697

May 1960

SUBJECT: METAL-CERAMIC EXTERNAL ANODE TUBES
INVESTIGATOR: Arnold H. March
CONTRACT: AF33(600)-38449, Bendix Aviation Corp.
ABSTRACT: On a previous contract, AF33(600)-26860, four metal ceramic tubes were developed which are capable of operation at high ambient temperatures in the vicinity of 500°C. The two tubes developed on Contract AF33(600)-38449 are to expand the previous group of four tubes to include a medium mu triode and a high mu triode.

AMC TR 60-7-697 (Continued)

Processing and construction are essentially similar for all six types except that the two tubes developed in the present contract utilize titanium plates as the active getter rather than the pressed ceralloy pill previously used.

Data presented includes results on high temperature life, vibration, shock, fatigue, heater cycling, and static characteristics. Processing and test specifications, as well as parts drawings, are also included.

AMC TR 60-7-732

June 1960

SUBJECT: LOW POWER SILICON VHF TRANSISTOR
INVESTIGATOR: P. P. Poliquin
CONTRACT: AF33(600)-39606, Radio Corporation of America
ABSTRACT: This abstract gives a brief description of the significant accomplishments of the Production Refinement, Low Power Silicon VHF Transistor Program (Contract No. AF33(600)-39606) by the Radio Corporation of America (Semiconductor and Materials Division) during the period from June 5, 1959 to June 5, 1960.

An NPN, all diffused, line structure design was chosen for this device because of its ability to operate at high frequencies and have a high collector-to-base breakdown voltage. This device, as designed, meets both the high frequency 250mc oscillator and amplifier requirements and the 100 volt VCBO specification.

High frequency silicon transistor manufacturing techniques were developed and refined to meet the production requirements of the contract. These techniques are shown to be suitable for large volume production. A description of the diffusion, photoresist, and metalizing operations are described in the section on Device Fabrication.

The contract specified an investigation of improved, and possibly automated, techniques and equipment to be built for the purpose of bringing the transistor manufacture up to mass production capabilities. RCA Somerville helped develop and bring into general use as an interim tool the Kulioke and Soffa bonding machine on which rates as high as 100 units per operator hour are anticipated. The Automatic Device Assembly Machine (ADAM) was designed and built. The machine is complete and operable, and has made transistors--both mechanically and electrically good--and a few samples have been delivered to the Electronic Technology Laboratory, WADD.

Transistors were fabricated and submitted as state-of-the-art samples.

SUBJECT: HIGH EFFICIENCY SILICON SOLAR CELLS
INVESTIGATOR: P. P. Poliquin
CONTRACT: AF33(600)-40497, Hoffman Electronics Inc.
ABSTRACT: Silicon photovoltaic cells for solar energy conversion can be produced in higher efficiency and varied configuration through the development of new and improved production techniques and processes.

The limitations of silicon solar cells are discussed and four major areas of improvement are considered: optimization of the p-layer thickness and series resistance; selection of crystals with improved properties; improved handling and fabrication techniques; decreased losses due to series resistance. Various studies and experiments made in order to obtain higher efficiency solar cells are detailed.

Studies are discussed which determined optimum junction depth and the diffusion parameters necessary to obtain these junction depths. Several diffusion experiments were performed and the variation of surface concentration, junction depth and the diffusion coefficient have been determined as a function of temperature. The effect of varying diffusion temperature on solar cell spectral response was investigated. Two methods of evaluating diffusions (junction depth and sheet resistance) are analyzed theoretically, and techniques for experimentally evaluating diffusion by these methods are discussed.

An improved plating technique which has significantly decreased cell series resistance and improved conversion efficiency is discussed. In order to further decrease cell series resistance, an evaluation to determine the optimum number of cell grid lines was made.

Technique variations such as ultrasonic cleaning and ultrasonic cleaning and hydrogen carrier gas for diffusion were studied.

Infrared transmission studies were made on silicon and silicon solar cells to determine whether design of cells for lower "in space" operating temperatures were feasible.

A light source simulating sunlight in space was developed. The sunlight simulator and various other parameters necessary to measure cells under space conditions are described.

The operation of a pilot production line using new techniques developed under this program is described.

Performance tests for the three one-square-foot panels required by this contract are detailed.

CHEMICAL ENGINEERING

AMC TR 59-7-436a

24 December 1959

SUBJECT: DEVELOPMENT OF TE-60 PLASTIC PARTS ASSEMBLY
INVESTIGATOR: Major Fritsch
CONTRACT: AF33(600)-31113, Thickol Chemical Corp.
ABSTRACT: Under the cognizance of the Manufacturing and Methods Division of the Air Materiel Command at Wright-Patterson Air Force Base, a program (Contract AF33(600)-31113) was conducted which demonstrated the feasibility of constructing chambers and nozzles for solid propellant motors from reinforced plastic materials. The T-61 (Matador) booster was selected for this program. A successful reinforced nozzle and chamber assembly was made of fiber glass roving and epoxy resin. This assembly weighed less than 600 lbs and when loaded, contained 1,450 lbs of T-35 (polysulfide) propellant. The performance of this motor was approximately 106,000 lbs of thrust for 2.6 seconds at 60°F. Six firings were made; all were successful. The firings were made at -30°, 60° and 160°F and included a drop test, a rough road test, and a temperature cycle test.

AMC TR 60-7-436b

April 1960

SUBJECT: DEVELOPMENT OF PLASTIC CASE FOR XM15 JATO ROCKET MOTOR (PLASTIC JATO CASE)
INVESTIGATOR: J. W. Hurst
CONTRACT: AF33(600)-31436, Rocketdyne Division
ABSTRACT: A XM15 JATO rocket motor case 50 percent lighter than a comparable steel case and competitive in cost has been fabricated from Epon 828 epoxy resin reinforced with helically wound glass fibers. Successful firings and storage tests confirmed the ability of the plastic case to meet all design requirements.

Sixty JATO motors were made of filament wound glass fiber reinforced plastic case, asbestos phenolic plastic aft head and nozzle, Buna-N rubber liner, and with the only metal parts being the forward head mounting bolts, rupture discs, steel insert for mounting the igniter assembly, and the mounting brackets. These steel parts accounted for 8 percent of the total weight. The comparable steel case weighs 94 pounds and costs \$37.00 per motor. The plastic motor case weighs 41 pounds (57 percent reduction) and should cost approximately \$37.00 per unit for a production rate of 5000 units per month.

AMC TR 60-7-632c

April 1960

SUBJECT: LARGE CERAMIC RADOME MANUFACTURE BY FLAME SPRAYING TECHNIQUE (CERAMIC RADOMES - FLAME SPRAYING)
INVESTIGATOR: D. C. Harleman
CONTRACT: AF33(600)-37877, Norton Company

AMC TR 60-7-632c (Continued)

ABSTRACT: Large ceramic (98.5% alumina) radomes for future hypersonic aerospace vehicles may possibly be fabricated using flame spraying, dry isostatic pressing or wet forming, hydrostatic pressing techniques.

Scale model (13" diameter x 39" high) radomes made by the flame spraying technique were fired successfully but only one thick wall radome had been ground successfully when the contract was terminated.

Norton Company's standard ROKIDE alumina composition was flame sprayed onto a rotating water cooled precision mandrel which had been coated previously with a thin layer of potassium chloride. After a sufficient thickness of alumina was deposited, the mandrel was suspended over a tank and the salt interlayer washed out with jets of water. When the salt layer had dissolved, the radome dropped free and was washed thoroughly to remove all traces of the salt. The radome was then dried at 85°C for 24 hours.

The radome blanks then were given a low temperature firing to 900°C to relieve stresses which had been developed in the spraying operation.

The final firing was accomplished in a large catenary gas-fired car kiln using special shrinkage setter plates. The radome blanks were fired in an upright position. The firing cycle took approximately one week. Firings were made to 1740°C., with a five-hour soak.

A large number of test specimens also were fabricated for physical and electrical testing to determine the properties of high alumina bodies. The test results are shown in the Final Technical Engineering Reports under Melpar, Inc., Contract AF33(600)-37908, AMC Project 7-632d, and Boeing Airplane Co., Contract AF33(600)-38574, AMC Project 7-632f.

AMC TR 60-7-632d

June 1960

SUBJECT: LARGE CERAMIC RADOME MANUFACTURE ELECTRICAL & MECHANICAL CHARACTERISTICS MEASUREMENTS (CERAMIC RADOMES--TESTING)

INVESTIGATOR: D. C. Harleman

CONTRACT: AF33(600)-37908, Melpar, Incorporated

ABSTRACT: Determinations were made of various electrical, mechanical, and thermal properties from 70°F. - 2500°F of two sets of three ceramic compositions for use in fabricating large ceramic (99.5 percent alumina) radomes for future hypersonic properties.

AMC TR 60-7-632d (Continued)

The experimental methods are discussed and data presented for the following properties:

- 1) Dielectric constant and loss tangent at X band
- 2) Tensile strength
- 3) Compressive strength
- 4) Flexural strength
- 5) Modulus of elasticity
- 6) Modulus of rigidity
- 7) Poisson's ratio
- 8) Specific heat
- 9) Normal total emissivity
- 10) Thermal diffusivity
- 11) Thermal expansion
- 12) Thermal conductivity

The samples tested were fabricated by:

- 1) Coors Porcelain Co., Golden, Colorado
Contract AF33(600)-37879, AMC Project 7-632a
- 2) Gladding-McBean Co., Los Angeles, Calif.
Contract AF33(600)-37952, AMC Project 7-632b
- 3) Norton Co., Worcester, Mass.
Contract AF33(600)-37877, AMC Project 7-632c

AMC TR 60-7-655

November 1960

SUBJECT: DEVELOPMENT OF MANUFACTURING PROCESSES FOR HIGH TEMPERATURE LUBRICANT AND HYDRAULIC FLUID BASE STOCKS

INVESTIGATOR: Charles Tanis

CONTRACT: AF33(600)-36237, American Potash & Chemical Corp.

ABSTRACT: This report describes the laboratory development work and successful pilot planting of a process for the manufacture of n-dodecyl tri(n-decyl) and n-hexadecyl tribenzyl silane fluid stocks.

On the basis of a literature survey, it was decided that the best route was to condense silicon tetrachloride with the appropriate alkylmagnesium halide. This process was developed in the laboratory to give an over-all yield of at least 30 per cent of tetraalkyl silane fluid of 95 percent purity. Analytical techniques were developed for following the various stages of the reaction, particularly through the use of infrared spectrum techniques.

A multi-purpose pilot plant was designed and constructed to manufacture these fluids. The laboratory processes were proven out successfully in the pilot plant. The capacity of this plant was 3 to 20 gallons per day, depending on product purity. In the pilot plant it was shown that a wipe film evaporator was the most versatile unit for rectifying these high boiling fluids. By the use of super-heated steam in connection with it, it was possible to separate trialkyl silane hydride and most of the hexaalkyl disiloxane impurities.

On the basis of the data collected in the pilot plant, a full-scale production unit has been designed. Specifications for raw materials, plant operation, etc., are included in a section of the report.

By means of a nine-stage process, bis(m-phenoxyphenyl) ether was prepared in very high purity and free of isomeric products in the laboratory. In five of these steps the yield was improved markedly over that previously reported in the literature. This resulted in a big reduction in cost.

AMC TR 60-7-719

December 1960

SUBJECT: DEVELOPMENT OF SILICON INFRARED OPTICAL COMPONENTS (TRANSMITTING WINDOWS)
INVESTIGATOR: Mr. D. C. Harleman
CONTRACT: AF33(600)-38085, Texas Instruments Incorporated
ABSTRACT: Silicon Plates up to 12 inches in diameter by 1 inch thick and silicon hemispherical domes up to 8 1/2 inches in diameter by 1/4 inch thick can be economically cast or segmented for use as infrared transmitting windows. Silicon vapor deposition techniques are not as feasible as casting. The optical transmission of nuclear reactor irradiated silicon showed some improvement for limited wavelengths.

A form of shell casting in which molten silicon freezes to a rotating silicon carbided graphite mandrel has been developed as a feasible and economical process to produce large size silicon domes and plates.

Silicon may be joined with an appropriate epoxy adhesive to produce suitable size mosaic structures. Plates 12 inches in diameter by 5/8 inch thick and domes 7-1/2 inch in diameter by 1/4 inch thick have been successfully produced. Size is limited by ability to fabricate suitable bonding fixtures.

AMC TR 60-7-719 (Continued)

For the sizes and shapes specified in the contract, casting is the more useful and economical process to use.

Silicon may be vapor deposited into 4 inch domes with a significant reduction in the absorption at 9 microns. Because of fabrication difficulties and a reduction in transmission between 4 - 8 microns, vapor deposition, as compared to cast silicon, is not presently a feasible production process.

The strength of cast and thermally deformed silicon is similar to the value obtained on grown silicon material. Typical values of Modulus of Elasticity and Rupture are 26,000,000 psi and 23,000 psi respectively.

A significant increase in the resistivity of silicon may be realized after irradiation. Little effect was found on the Silicon Monoxide coating before and after irradiation. Irradiating low resistivity silicon decreases the silicon absorption coefficient the greatest in the 5 to 6 micron range. Some of the irradiation induced defects which reduce the room temperature absorption coefficient are annealed out at temperatures about 400°C.

FABRICATION

AMC TR 60-7-110d

July 1960

SUBJECT:

NUMERICALLY CONTROLLED MILLING MACHINE

CONTRACT:

AF33(038)-24007, Servomechanisms Laboratory,
Massachusetts Institute of Technology

ABSTRACT:

Numerical control is a design technique which extends the benefits of automatization to moderate-volume manufacturing processes previously limited to manual production methods. A milling machine, now in operation at the Massachusetts Institute of Technology, illustrates the technical feasibility of numerical control and, on the basis of initial test operation, shows promise of becoming a valuable production tool.

A new concept of automatic machine-tool control now being demonstrated at the Servomechanisms Laboratory at M.I.T. promises to simplify some of the manufacturing problems of medium and small run production often encountered in the aircraft industry.

Called numerical control, this technique has been applied by M.I.T., under the sponsorship of the Air Materiel Command of the U.S. Air Force, to a milling machine controlled by a numerical code punched on paper tape.

Since March 1952 when construction was completed, the milling machine has been used as an experimental production unit to evaluate the economic aspects of numerical control and as a basis of a study of the application of numerical control to other machine tools and manufacturing processes of particular interest to the aircraft industry.

While a complete evaluation will be possible only after the present program of trial operation has been carried much further, it is already clear that the numerically controlled milling machine is a valuable production tool which combines high productivity, flexibility, and precision. The control system, during approximately 4,000 hours of operation, has exhibited a high degree of reliability. Operational experience shows that the system requires as little maintenance as the basic machine tool.

AMC TR 60-7-250a

March 1960

SUBJECT:

DEVELOPMENT OF FLASHWELDING TECHNIQUES FOR NON-FERROUS ALLOYS

INVESTIGATOR:

B. E. Price

CONTRACT:

AF33(600)-22246, Lockheed Aircraft Corp.

ABSTRACT:

Flashwelding techniques and machine set-up data curves have been developed, for 6061, 2014, 2024 and 7075 aluminum alloys which produced flash weldments possessing acceptable quality, ductility and fatigue life. The static strength levels of developed

AMC TR 60-7-250a (Continued)

welds were equal to or greater than the minimum strengths of the parent metal. A proposed Military Specification is presented.

The fatigue strength of 6061, 2014, 2024 and 7075 weldments were substantially the same with a strength reduction approximately equivalent to a stress concentration factor K_t of 3.0 when compared with basic NACA data on comparable aluminum alloy sheet specimens. Flushing of the weld and then shot peening resulted in a fatigue strength comparable to the parent metal.

Standard protective finishes common to bare aluminum alloys will satisfactorily protect weldments from pure corrosion. However, the higher strength aluminum alloys, 2024, and 7075, were susceptible to stress corrosion.

The primary factor responsible for the high stress corrosion susceptibility of 7075-T6 flash weldments is the short transverse grain orientation formed in the weld as the result of upsetting during flashwelding. Comparative studies of the effects of grain orientation in non-welded 7075-T6 reveals that the stress corrosion of 7075-T6 flash weldments is defined by the short transverse stress corrosion properties of the parent metal. Variations in the flash welding process, post weld thermal treatments, or use of chemical conversion coatings provide no significant improvement in stress corrosion resistance. The combination of post weld shot peening and aging affords improvement but does not prevent stress corrosion cracking.

The short transverse grain orientation of the weld in 2024-T4 flash weldments is not the limiting factor determining stress corrosion susceptibility. A significant improvement in stress corrosion resistance and strength can be obtained by aging 2024-T4 flash weldments six or more hours at 370°F after flash welding and by a combination of post weld shot peening and aging.

Post weld aging of 2024 flash weldments, supplemented by protective coatings and shot peening, is recommended as a means of insuring maximum serviceability in structural applications where stress corrosion is a consideration. The use of 7075 flash weldments for structural components in corrosive environments or for extended service life applications is not recommended.

AMC TR 60-7-488

March 1960

SUBJECT: DEVELOPMENT OF A.C. INVERTER STATOR WINDING MACHINE
CONTRACT: AF33(600)-33405, American Machine & Foundry Co.
ABSTRACT: Phase I was a study program for the development of an automatic AC inverter stator winding machine. Patents, existing winding machines, and techniques used by the rotary electrical industry

AMC TR 60-7-488 (Continued)

were investigated. With an appraisal of inverter characteristics, specifications for the machine were established.

Phase II was a design program to develop a machine to meet the requirements of the specifications of Phase I. After the jury rig testing of two concepts, a design was evolved for the prototype machine. Components consisted of a Wire Feed System, End Loop Forming System, Translating Mechanism, Skewing Mechanism, Oscillating Mechanism, Chuck and Indexing Mechanism, Motor and Drive System and Counting, Programming and Speed Control.

Phase III called for the final development of the components, the fabrication assembly, testing and the evaluation of the machine. The prototype machine was built to wind 250 VA uniform stators. A detailed description is given of the machine.

Phase IV provided for the modification of the Machine to wind 750 VA concentric stators. To program this pattern of winding, modifications were made to the arbor drive, the nozzle drive, the coil span mechanism, the shoe coil forming device and the wire retention device.

The machine successfully produced uniform wound 250 VA size and concentric wound 750 VA size stators.

AMC TR 60-7-561

December 1960

SUBJECT: EVALUATION OF NEW TITANIUM SHEET ALLOYS FOR USE IN AIRFRAME CONSTRUCTION
CONTRACT: AF33(600)-33597, North American Aviation, Inc.
ABSTRACT: Descriptions of the basic objectives of Contract AF33(600)-33597, the selection of parts to be fabricated under this contract, the selection of the sheet alloys to be evaluated, and the results of a preliminary evaluation of the Ti-16V-2.5Al and Ti-4Al-3Mo-1V sheet alloys are presented in this volume.

Based on a survey of the available data on properties and on production status, the Ti-16V-2.5Al, Ti-4Al-3Mo-1V, and Ti-3Al-6Mo alloys were selected for evaluation. Before any extensive tests could be made, production of the Ti-3Al-6Mo was discontinued, and the comparative evaluation was restricted to mechanical properties and forming characteristics of the two remaining alloys.

The average short-time tension and compression strengths from room temperature to 900°F of the Ti-16V-2.5Al alloy in the aged condition was higher than the average strengths of the Ti-4Al-3Mo alloy. Based on the data obtained, the Ti-16V-2.5Al would have design tensile minimums (2) of 153,000 psi yield and the 165,800 psi

AMC TR 60-7-561(Continued)

ultimate versus 147,000 psi yield and 184,600 psi ultimate strengths for the Ti-4Al-3Mo-1V alloy. The Ti-4Al-3Mo-1V alloy had higher tensile elongation and higher modulus of elasticity (E) than the Ti-16V-2.5 Al alloy. A limited number of creep tests indicated that the Ti-4Al-3Mo-1V alloy would have greater tensile creep resistance at 600°F and 800°F than the Ti-16V-2.5Al alloy.

Straining the Ti-16V-2.5 Al alloy in the solution-treated condition followed by aging, resulted in a substantial loss in tensile strength. The tensile strength of the Ti-4Al-3Mo-1V alloy increased when strained and aged.

The surface condition of the sheets (grind marks, scale pits, and other defects) had a major effect on the results of the forming tests, particularly on the bend radii. Both alloys had a range of approximately 3.5T to 5.5T bend radius. Bends made in the Ti-16V-2.5Al alloy had much greater spring back than bends made in the Ti-4Al-3Mo-1V alloy.

Difficulty was experienced in joggling the Ti-16V-2.5Al alloys. Larger runout depth-to-length ratios were required to joggle the Ti-16V-2.5 Al alloy than were required to joggle the Ti-4Al-3Mo-1V alloy.

Greater local stretch and shrink values were obtained in the Ti-4Al-3Mo-1V alloy than in the Ti-16V-2.5Al alloy in both hydropress and stretch forming operations.

After reviewing with the Air Force the mechanical properties and forming data obtained on the two alloys, the Ti-4Al-3Mo-1V alloy was selected to be used for the fabrication of parts and the complete metallurgical evaluation as described in subsequent volumes of this report.

AMC TR 7-618

November 1959

SUBJECT: TITANIUM SANDWICH PANEL RESEARCH & DEVELOPMENT
CONTRACT: AF33(600)-34392, Convair
ABSTRACT: Five titanium alloys (6Al-4V, 16V-2.5Al, 3Al-6Mo, 4Al-3Mo-1V, and RS-140) and numerous braze alloys and adhesives were evaluated for suitability for structural sandwich construction in the temperature range of -67° to 800°F. RS-140 was chosen as the prime alloy because of superior properties and availability. 16V-2.5Al and 6Al-4V were selected as "back-up" alloys. Extensive mechanical property testing of RS-140 was accomplished.

Three brazed and two bonded production-size sandwich panel designs were selected for developmental fabrication and testing. These were modifications of B-58 stainless steel and aluminum sandwich panel designs.

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Procedures for brazing and bonding were developed. A new and superior braze alloy was developed. Preliminary test data on small sandwich specimens was obtained. Limitations of fabrication by drawing, bending, machining, and welding were tentatively established.

Testing of the brazed structural test panels showed that they possessed excellent structural efficiency at temperatures from -67°F to 800°F. Strength-to-weight ratio of brazed sandwich panels construction used in the B-58 by a factor of approximately 1.50.

AMC TR 60-7-627

July 1960

SUBJECT: STAINLESS STEEL SANDWICH COMPOSITE MATERIAL PROGRAM
CONTRACT: AF 33(600)-35074, Sciaky Brothers, Inc.
ABSTRACT: This is the final report of a research program to develop tooling methods and processes for resistance welding of stainless steel sandwich composite material. The program was conducted by the Sciaky Bros., Inc. Research Division, 2311 Purdue Ave., Los Angeles 64, California, for the Air Materiel Command.

The program included extensive investigation of core configurations; for ease of manufacture, accessibility of weld tooling, structural aspects. Basic resistance weld methods, weld tooling, and electrical components used were of completely standard design or slight variation. The findings of the above were resolved to parameters which culminated in the construction of two experimental sandwich welding machines.

Instrumentation and testing equipment were commercial. Tests conducted were per MIL-STD-401A, MIL-C-21275 and with consideration given the substance of ATC Report No. ARTC-17 and FMS-003(c).

The feasibility of resistance welded stainless steel sandwich composite material has been demonstrated.

AMC TR 60-7-681

November 1960

SUBJECT: SUB-ZERO MACHINING PROGRAM
CONTRACT: AF33(600)-38296, Convair
ABSTRACT: The application of sub-zero coolants to machining high-strength, high temperatures alloys will, in certain applications, increase metal removal rates up to 400%. These increases are not necessarily concurrent with cost reduction, because of the additional expense of sub-zero equipment, coolants and machine maintenance. The mechanical properties of the materials tested were not affected by sub-zero temperatures.

Cost comparisons showed sub-zero coolants to be

AMC TR 60-7-681 (Continued)

more economical in only one of the four tests when the machining of production parts was simulated. The results indicated that uninterrupted cutting operations on long cuts can be accomplished more efficiently with sub-zero coolants than with conventional coolants. Machining with sub-zero coolants is uneconomical on short runs and in interrupted cutting operations.

The three sub-zero cooling methods evaluated were:

1. Application of CO_2 mist at the cutting edge (-81°F to -101°F).
2. Flooding the workpiece and tool with ABCO 156 A Solvolene (-58°F)
3. Freezing the workpiece in a cold chest (-110°F) prior to machining.

The machining operations performed were turning, milling, drilling, tapping and grinding. The materials used in the tests were:

A-286, an austenitic stainless steel
L-605, a cobalt base alloy
R-235, a nickel base alloy
H-11, a hot-work die steel
Mo-0.5Ti, a molybdenum base alloy

Room temperature tests were performed with conventional coolants to establish tool geometry, tool material, feed and cutting speed for comparison with sub-zero machining tests. The results are presented in graphical form.

AMC TR 60-7-765

November 1959

SUBJECT: RESISTANCE HEATING OF TITANIUM AND TITANIUM ALLOYS
CONTRACT: AF33(600)-38042, Republic Aviation Corp.
ABSTRACT: An investigation aimed at determining the variables, equipment requirements and control problems of the resistance heating of titanium was conducted. This program, at its outset, determined that temperature control was the primary problem. The one plausible solution appeared to be control through radiation pyrometry. Although accurate correlation of emissivity of bare titanium to temperature proved impossible because of considerable surface condition change, the program was brought to a successful conclusion through the use of stable emissive coatings on the workpiece.

AMC TR 60-7-766

November 1959

SUBJECT: FORMING, HEAT TREATING AND MACHINING OF 5-Cr-Mo-V TOOL STEELS
CONTRACT: AF33(600)-38042, Republic Aviation Corp.
ABSTRACT: A typical detailed evaluation of the 5-Cr-Mo-V

AMC TR 60-7-766 (Continued)

tool steel revealed the design criteria covering the application of this material in airframe construction. This evaluation, in its extensive breadth, covered all of the standard forming methods, marking methods, heat treatment procedures and the machining of this steel in the sheet and cast state. The resultant data provides an excellent foundation for extensive use of the material in sheet form and will assist in alleviating many initial production problems.

AMC TR 60-7-767

November 1959

SUBJECT: FORMING OF INCONEL X
CONTRACT: AF33(600)-38042, Republic Aviation Corp.
ABSTRACT: The basic criteria for the forming of Inconel X into typical airframe parts has been determined. In addition to studies of the basic fundamentals, investigations uncovered significant features involved in the initial heat treatment and the processing through some of the more sophisticated manufacturing operations. The results clearly indicate that this material is readily usable if certain precautions peculiar to this material are observed.

AMC TR 60-7-768

November 1959

SUBJECT: BERYLLIUM COPPER DIES
CONTRACT: AF33(600)-38042, Republic Aviation Corp.
ABSTRACT: The non-oxidizing and non-galling properties and superior thermal conductivity of beryllium copper have been exploited by using this material for aircraft forming dies. This program has established the great potentials of integrally heated cast beryllium copper tooling. This tooling has found application on drop hammer, hydropress, power brake, power press and stretch press operations. One of the primary advantages is incorporated in the perfected precision casting process and the resultant economics.

AMC TR 60-7-769

November 1959

SUBJECT: TUNGSTEN INERT GAS ARC WELDING OF HIGH STRENGTH STEELS
CONTRACT: AF33(600)-38042, Republic Aviation Corp.
ABSTRACT: The procedures for the fabrication of reproducible welds of aircraft quality of 5-Cr-Mo-V and AM-355 steels have been established. Both steels are readily weldable by conventional techniques if certain well-defined steps in the procedure are followed. These steps involve specific thermal processing post and prior to the welding. The design criteria established excellent weld efficiencies at high heat treatment levels (250-300 ksi).

AMC TR 60-7-770

November 1959

SUBJECT: METALLIC ARC WELDING OF HIGH STRENGTH STEELS
CONTRACT: AF33(600)-38042, Republic Aviation Corp.
ABSTRACT: A number of commercial coated electrodes were evaluated for the aircraft quality welding of 5-Cr-Mo-V and SAE 4340 alloy steels. Several of these electrodes prove satisfactory in producing weldments meeting the requirements of the applicable military specifications. Extensive testing clearly defines the various properties peculiar to each coated electrode in comparison to specified values. This data will be met by enthusiastic acceptance throughout the airframe industry in that it is immediately applicable to production problems on hand.

AMC TR 60-7-807

May 1960

SUBJECT: BERYLLIUM FASTENER PROGRAM
INVESTIGATOR: E. F. Gowen, Jr.
CONTRACT: AF33(600)-39728, Standard Pressed Steel Co.
ABSTRACT: A program to determine the feasibility of manufacture of beryllium fasteners for aircraft and missile structural applications was made possible through the support and sponsorship extended by the Manufacturing Methods Division, LMBM, AMC Aeronautical Systems Center.

Since beryllium has a weight less than one-fourth an equal volume of steel, substantial weight savings could be realized by employing beryllium in fasteners. Therefore, it was desirable to learn as quickly as possible how beryllium fasteners could be manufactured and what their mechanical properties would be.

This information was obtained in a two step program. At first, the bolts were produced to a current configuration by standard manufacturing techniques. From the data and experience obtained, changes in design and manufacturing methods were employed and evaluated to further improve the potential of beryllium bolts.

It was found necessary to establish special controls for inspection of incoming bar stock. Current methods were inadequate to insure suitable materials.

Because the currently available beryllium material is extremely notch sensitive, modifications of existing bolts designs were found necessary to enhance the properties of the fasteners. These included the use of large radii in the thread roots and special surface treatment of the finished bolts.

Standard methods of fastener manufacturing were found applicable to the production of beryllium bolts. The only additional precautions necessary were the addition of exhaust systems when machining or grinding the parts. These collected all particles of beryllium oxide so that machine operators were not exposed to these toxic materials.

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The data shows that beryllium shear fasteners can be produced with a great advantage in their strength-to-weight ratios over current fasteners fabricated from both titanium and high strength steel. Shear and fatigue strengths were 70,000 psi and 40,000 psi respectively. The strength of the base material was a minimum of 85,000 psi. Because of its high degree of notch-sensitivity, beryllium is not recommended for tension fasteners.

AMC TR 60-7-755

December 1960

SUBJECT: FORGING PROGRAM TO IMPROVE MECHANICAL PROPERTIES OF LARGE STEEL FORGINGS

INVESTIGATOR: Mr. G. L. Campbell

CONTRACT: AF33(600)-38060, Ladish Company

ABSTRACT: Statistical analysis of 5,000 test data from nine forgings of AISI 4340 aircraft quality steel for a landing gear component shows that transverse ductility and toughness decreases (1) as a function of distance from surface to center of the ingot, (2) as a function of the hardenability of the particular ingot heat studied, and (3) as a function of the amount of reduction in cross-section when forging the ingot beyond a four to one reduction up to an eight to one reduction. Transverse properties are more affected than longitudinal properties and the effects are more pronounced as the strength level increases in the range of 200,000 - 300,000 pounds per square inch.

Steel manufacturers have pushed the tensile properties of 4340 and other steel alloys to levels approaching 280,000 pounds per square inch. In general, satisfactory longitudinal properties have been shown but these are not matched in the transverse direction.

Conventional steelmaking practice and forging result in parts with a large differential between longitudinal and transverse mechanical properties, with transverse, particularly ductility, being lower. A large amount of AISI 4340 aircraft high strength steel has been made under these conditions but no thorough critical evaluation incorporating a number of controlled variables has ever been made with the objective to improve the mechanical properties of forgings made from this steel.

To minimize the adverse property effects resulting from melting and freezing phenomena supplementary to the forging, two heats of steel were made utilizing the best melting and processing practices. Two ingot sizes were reduced to 12 inch RCS billets for evaluation, including the following tests: macroetch, grain size, micro-cleanliness, magnetic particle cleanliness, chemical analysis, tensile and hardenability. Landing gear component forgings made from the billets were further processed, sectioned and tested, based upon the effects of forging reduction ratio, hardenability, strength level, the test direction and location upon strength, ductility and toughness.

BASIC INDUSTRY

AMC TR 60-7-257

July 1960

SUBJECT: DEVELOPMENT OF AN EXTRUSION METHOD FOR THE PRODUCTION OF WIDE PROFILE EXTRUSIONS
INVESTIGATOR: T. S. Felker
CONTRACT: AF33(600)-21525, Loewy-Hydropress Division
ABSTRACT: Wide, flat, thin-profile aluminum extrusions for high performance aircraft and missile application can be readily and economically extruded by means of a rectangular (flat) billet container in conventional extrusion presses rather than being extruded from round containers in "V" or round shapes requiring subsequent flattening.

A rectangular container proved superior to the round container in that thinner sections are possible and greater unit pressure effected more efficient metal flow. The necessity for converting the extruded section from "V" or other shapes which resulted from previously used round containers was eliminated.

In use, the rectangular container made possible an extrusion pressure of 109,000 psi on the billet. An integrally stiffened panel of 7075 aluminum alloy, 18 inches wide, with web thickness of 0.037 inches, having eight stiffeners, each one inch in height by 0.100 inches thick was successfully extruded to a length of 25 feet and with excellent surface condition.

Specific die entrance angles and sturdier die design were necessary to withstand the higher extrusion pressures.

AMC TR 61-7-557

March 1961

SUBJECT: DEVELOPMENT OF IMPROVED METHODS FOR COLD EXTRUSION OF TITANIUM
INVESTIGATOR: R. J. Fiorentino, A. M. Sabroff, P. D. Frost
CONTRACT: AF33(600)-33540, Battelle Memorial Institute
ABSTRACT: Precision hollow shapes of unalloyed titanium and Ti-3Al-2.5V alloy can be backward extruded at 50 percent reduction at practical working pressures, using a fluoride-phosphate coating and properly designed punches. Applications of the process to fabrication of a titanium MS-21921 hexagonal nut for aircraft flareless tube fittings shows a potential saving of 48.5 percent of the material and labor costs over automatic screw machine techniques alone.

The contour of the optimum punch tip is described by the segment of a circle having a half-angle of 70 degrees. Backward extrusion pressures for unalloyed titanium are 340,000 psi with the optimum punch shape, compared with 400,000 psi required by a flat punch.

The hexagonal nut was fabricated by first backward

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extruding a solid cylindrical billet, 1 inch in diameter x 5/8-inch long, into a full-sized hexagonal cup, 1 inch across the flats x 25/32-inch-diameter bore x 1 inch long. The cup is then threaded and finished on an automatic screw machine.

The potential extrudability of five commercial titanium alloys at 80,500, and 1000°F was estimated from compressive flow-stress curves. A method is suggested for estimating the backward-extrusion pressure requirements for 50 percent reduction, knowing the compressive flow stress at this same reduction.

Forward-extrusion studies on the all-beta Ti-13V-11Cr-3Al alloy showed it can be cold extruded into solid rounds at reductions of 20, 40, and 60 percent. Pressures over this range were from 124,000 to 252,000 psi, about 60,000 to 80,000 psi greater than those for the same reductions on unalloyed titanium.

AMC TR 60-7-577

July 1960

SUBJECT: EXTRUSION DIE DEVELOPMENT, TITANIUM AND STEEL
INVESTIGATOR: Mr. T. S. Felker
CONTRACT: AF33(600)-36303, Harvey Aluminum, Inc.
ABSTRACT: Precision hot extrusion of titanium and steel structural sections for aircraft, missiles, and aerospace vehicles may be accomplished most effectively with bonded carbide or M-36 high speed steel dies and a lubricant of the graphite-moly-disulphide type.

In order to adequately evaluate many potential die materials, a testing device was designed and constructed. This machine simulated, on a laboratory basis, extrusion conditions which are normally encountered by use of a conventional hydraulic extrusion press. The cost per test was thus greatly reduced. Subsequently, extrusions were produced in limited numbers using the press to prove the validity of the evaluations secured on the laboratory device.

The experiments were conducted using a titanium alloy (2.5 Al - 5 Sn) as a flat surface heated to 1800°F. This plate simulate the billet in the extrusion process. Various die materials in the form of a nib with a 3/4 inch sphere at the tip were tested, each heated to 750°F. Die materials were thus evaluated by moving the nib over the plate under controlled loading and speed and accurately measuring the scoring produced.

Approximately 50 die materials were tried in this effort. These included carbides, cermets, ceramics, steel alloys, and refractory alloys. In addition, numerous types of die surface treatments were evaluated by these simulated tests.

SUBJECT: HIGH ENERGY RATE METAL FORMING
INVESTIGATOR: F. Pipher, Glen Rardin, W. Richter
CONTRACT: AF33(600)-35543, Lockheed Aircraft Corp.
ABSTRACT: High energy rate metal forming techniques, for fabrication of structural high strength components for airframe and spacecraft sheet metal applications are presented. The forming techniques developed demonstrate that the controlled application of the energy available from high velocity explosives is a safe, efficient and economical production forming method. The inherent advantages, as well as the limitations of this new forming method, are presented for consideration in the design and production of "difficult to form" sheet metal components.

Explosive forming techniques were developed for five (5) different configurations. Multiple operations were employed as required by the material used and the severity of the forming operations. High velocity explosives were used throughout the production phase of this program. Descriptions of the explosives and methods for preparation of cylindrical, linear and shaped charges are presented. Metallurgical, stress corrosion and mechanical property data is included. Tank design criteria were established.

General tool design recommendations are presented which should further improve the cost advantage of explosive forming.

SUBJECT: DEVELOPMENT OF BERYLLIUM SHEETS ROLLED FLAT TO GAUGE
INVESTIGATOR: K. Wikle
CONTRACT: AF33(600)-35829, Brush Beryllium Co.
ABSTRACT: Beryllium sheet material for high performance aircraft, missiles and orbital vehicle application can be rolled to specified thickness, flatness and up to 24" x 60" in size under controlled mill rolling and flattening techniques.

Beryllium sheet produced by the developed process was successfully fabricated into typical aerospace components, thereby establishing beryllium as an acceptable engineering material because of the improved ductility demonstrated.

Conventional rolling in the direction of the longitudinal axis proved insufficient to produce desired mechanical properties for aeronautical application, but consistent mechanical properties within 10% in both the longitudinal and transverse directions were obtained by the developed cross rolling technique for the material.

Minimum cost and the mechanical properties required for the sheet material dictated the use of 200 mesh, vacuum hot pressed, QMV beryllium as the starting block material, rolling at 1400 - 1500°F., followed by flattening between platens.

SUBJECT: IMPROVEMENT OF MECHANICAL PROPERTIES OF STEEL CASTINGS BY PRESS FORGING

INVESTIGATOR: D. C. Harleman

CONTRACT: AF33(600)-36387, Armour Research Foundation

ABSTRACT: Steel castings for high performance aircraft and missiles were markedly improved in strength by the application of a press forging operation accomplished under controlled conditions of temperature and deformation.

The most suitable alloys for the technique are low-carbon, high-alloy types of steel where the martensite can sustain deformation. The casting alloys of this type investigated were AMS 5363A (an 18-8 stainless), and 17-4 PH stainless steels. In the former, strength increases of 400 - 600% in yield strength and 200 - 250% in UTS were obtained by press forging at -100°F with 40% deformation, followed by tempering at 650°F. In the case of the 17-4 PH steel, increases in both yield and UTS of approximately 10% were obtained by deforming the predominantly martensitic structure 10% at room temperature followed by aging at 860°F.

SAE 4340 is responsive to strengthening by warm working, prior to transformation and at the 240,000 psi strength level; the yield point can be increased by 15 percent and the UTS by 10 percent. The composition of 4340 is not entirely suited to these techniques.

Utilization of the process with the low-carbon, high-alloy steels is feasible but not without certain difficulties. These involve the design of closed dies of adequate strength to press forge at the lower temperatures. Selective press forging of critical areas in castings by semi-open die techniques, is perfectly feasible.

SUBJECT: BERYLLIUM CASTING

INVESTIGATOR: A. H. Langenheim

CONTRACT: AF33(600)-37902, Beryllium Corporation

ABSTRACT: The objective in Phase II of this contract was to develop a reliable technique for producing fine grained, sound beryllium cast ingots. Four approaches to obtain grain refinement were evaluated including; alloying additions, inoculation, mold vibration, and accelerated cooling through mold design. Of these, the latter was the most effecting in achieving sound, fine grained ingots.

The alloying additions in varying amounts of lanthanum, zirconium, germanium, and silver were evaluated for grain refinement effects. Five ingots were poured with lanthanum additions of 0.07% to 0.5%. Ingot XP-183 with 0.07% lanthanum produced the lowest average grain size of 0.115 (mm) in this series. Thirteen ingots containing zirconium additions from 0.15% to 2.43% were evaluated.

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Ingot XP-186 in this group with 0.27% zirconium had a low average grain size of 0.097 (mm). Eleven germanium alloy castings were poured with germanium concentrations of less than 15 ppm to 0.6%. In this series Ingot XP-96, having 0.13% germanium, had the lowest average grain size of 0.101 (mm). The silver alloy series included six castings. The amount of silver additions varied from 0.23% to 16.1% with ingot XP-89 (4.94% silver) having the lowest average grain size of 0.137 (mm). In summary of the data presented, the zirconium alloy containing 0.27% zirconium, poured at 1350°C into a cold 1 1/2" diameter mold produced the lowest average grain size.

Other alloying elements including aluminum, titanium, silicon, and silver were used either singly or in combination to evaluate their effect on surface finish, apparent fluidity of the metal, and ingot soundness. Fifteen heats were poured. The silicon and titanium in combination produced good fluidity and sound castings were produced using a combination of titanium, silicon, and silver.

Tantalum nitride and tungsten carbide were found to be ineffective as inoculants, in the concentrations obtained in the eighteen melts poured.

The use of a low frequency mechanical vibration of the mold produced grain refinement in six ingots poured. The study suggests that a higher energy in either frequency or amplitude would be beneficial in breaking up the columnar grains as they grow during solidification.

The use of heavy walled molds made of high thermal conductivity materials to control solidification rates was most effective in achieving sound, fine grained ingots.

Three fine grained ingots were extruded at a reduction ratio of 4.5:1. The first ingot containing 0.76% silver was poured at 1300°C. The second contained 1.22% silver and was poured under the same conditions. The third ingot was unalloyed beryllium poured at 1400°C. No vibration was used on the molds of these ingots. Sections of the first two extrusions were clad and subsequently rolled to 0.090 sheet. Metallographic examination of the sheet indicated that a recrystallized structure can be produced which is equiaxed and has a grain size comparable to hot pressed beryllium powder.

AMC TR 60-7-656

September 1960

SUBJECT: DEVELOPMENT OF TITANIUM ALLOY CASTING METHOD
INVESTIGATOR: A. H. Langenheim
CONTRACT: AF33(600)-36450, Boeing Airplane Co.
ABSTRACT: A commercially feasible process was developed for production of titanium alloy castings for high performance aerospace

AMC TR 60-7-656 (Continued)

vehicle applications. The recommended practice utilized a vacuum consumable-electrode arc furnace, water cooled copper, tilt-pour crucible, expendable rammed graphite molds, and centrifuge casting techniques. The Ti-6Al-4V alloy is the best casting alloy presently available, with as-cast tensile strength in excess of 150,000 psi and excellent surface finish.

A vacuum, consumable-electrode, arc furnace with water-cooled copper, tilt-pour skull crucible is suitable for melting and pouring of production heats of titanium alloys. Melting stock and electrodes were prepared by inert-gas shielded arc welding compacted sponge and re-cycled solid scrap. An expendable rammed graphite mold has been found most suitable for production casting of titanium alloys. Graphite base shell molds and investment molds were partially developed and offer considerable potential for future investigation.

Feeding characteristics were determined in rammed and machined graphite molds. In general, increasing the amount of taper improves feeding distance, and the relationship between taper and section thickness was established and tabulated. Centrifuge casting did not significantly change feeding characteristics but did help in filling out thin sections in the mold. A satisfactory heat treatment to improve the properties of cast Ti-6Al-4V alloy was attempted, but the best properties were obtained in the as-cast condition.

Satisfactory finishing operations include, removal of gates and risers by power saw or abrasive cut-off wheel, grinding and belt sanding for primary clean up, and sand blasting for final clean up. A satisfactory acid solution and processing technique was developed for chemical removal of contaminated surface material. For best fatigue properties, up to 0.015 inch per surface should be chemically removed, using the solution and process developed.

Effects of interstitial and alloying elements were determined and composition limits established. Elevated temperature tensile properties were determined. Selected shapes and sizes were successfully developed and produced, and compared with the corresponding production components by structural testing. Statistically valid minimum design allowables were established and minimum dimensional tolerances for design purposes were determined. Fundamentals of casting design do not differ greatly from principles used for other metals.

AMC TR 60-7-667

December 1960

SUBJECT: EVALUATION OF NUMERICALLY CONTROLLED MACHINING OF
FORGING DIES
CONTRACT: AF33(600)-36136, Harvey Aluminum, Inc.
ABSTRACT: The use of a numerically controlled milling and

AMC TR 60-7-667 (Continued)

profiling machine has proven practicable and feasible in the machining of dies, templates, forgings, and Elox Electrodes of aircraft quality. Compared to conventional methods of processing the major benefits attained are:

- a. Closer tolerances (- .0005") are obtained through numerical control.
- b. Fine finishing of die surfaces requiring little or no polishing.
- c. Exact duplication of a die to the original dimension when a re-sinking operation is required, by use of the original tape.

This evaluation program developed the use of and evaluated the operation of a Kearney & Trecker Profiler in conjunction with the Bendix Corp. Compac 01 Numerical Control.

During this evaluation program it was found that when more than one die of the same configuration is required, the economics are definitely in favor of numerical controlled die sinking. The programming and tape preparation time for making a single die is approximately twice that of machining time. After a tape has once been made, additional dies can be sunk on a comparatively rapid basis.

The numerical controlled machine can also be used economically and interchangeably for sinking dies, machining forgings, machining templates, electrodes, and any combination of two or more of the above to secure an economical forging, despite intricate configurations. Many of these combinations could be used to manufacture forgings in support of the Air Force programs such as window frames, landing gear support forgings and a variety of other forgings for aircraft work. This type of forging can be manufactured faster and at a lower cost because of usage of the numerical controlled machine and will result in lower costs to the Air Force.

This method of sinking forging dies has proven to be very accurate, but the system does have some economical and practical limitations. Competitive requirements, however, may require all manufacturing operations to be performed by numerical controlled machines, which will lead to better products produced at less cost and with less overall time.

AMC TR 60-7-667a

September 1960

SUBJECT: EVALUATION OF NUMERICALLY CONTROLLED MACHINING OF FORGING DIES
INVESTIGATOR: D. C. Harleman
CONTRACT: AF33(600)-36176, Wyman-Gordon Company
ABSTRACT: Numerically controlled machining of forging dies provided a superior finish and greater accuracy as compared to con-

AMC TR 60-7-667a (Continued)

ventional methods of die sinking. Outstanding results were obtained on interim work performed on templates and the machining of parts. Repeatability was excellent and results proved more satisfactory as to accuracy, finish, and time when compared to conventional methods.

The continuing development of programming techniques, specifically the mode of communication between the human element and the machine, to translate drawing information into control tapes which direct the tool aided in moving from simple shapes to more complex configurations. The establishment of control tape centers where the burden of calculations is transferred to computers expedited the generation of control tapes. Use of automatic programming systems of "systematized solutions" reduced programming time to a period competitive with conventional models and masters.

During the life of this contract, seven die blocks were programmed. One of these die blocks was an experimental impression designed to initiate the numerical control group into the intricacies of programming and to require the machine to perform in various operational modes. The other six blocks machined were production forging dies capable of producing finished forged parts. Only finish die blocks, for which the impressions could be mathematically defined, were programmed and machined. Former or blocker dies, which have blended or "faired" surfaces, were not attempted because computer routines for such work were not available.

Operational problems posed no particular difficulties after the normal break-in period. Hydraulic and electronic maintenance routines were established after a thorough personnel training. Maintenance costs were considered consistent with this type of equipment and were not excessive.

AMC TR 60-7-670

November 1960

SUBJECT: DEVELOPMENT OF COATINGS FOR TITANIUM DURING MILL PROCESSING

INVESTIGATOR: M. A. Komertz

CONTRACT: AF33(600)-37425, Crucible Steel Co. of America

ABSTRACT: Three proprietary ceramic-type coatings to protect and reduce the contamination of aircraft and missile quality titanium alloy sheet during rolling mill processing were determined as satisfactory. These coatings are effective in air and in furnace atmospheres at temperatures up to 2000°F for periods of four hours or more and act as lubricants during mill rolling.

Coatings presently available were not designed to withstand forging and rolling operations so evaluation and development were necessary under simulated conditions. Coatings for titanium alloys prevent rapid oxidation in air or in furnace atmospheres at high tempera-

AMC TR 60-7-670 (Continued)

tures required for reheating, forging, rolling and heat treating, thus preventing high metal losses through scale formation and metal contamination and minimizing surface conditioning costs. This investigation included the testing of twenty-one proprietary coatings and forty-one laboratory mixes of ceramic and non-ceramic types. The coatings showing the most promise were tested at temperatures up to 2300°F. These coatings, Markal Company's CRT, du Pont's J-400, and Crucible's No. 50 were chosen for hot rolling experiments under simulated mill conditions. Coated and uncoated sheet bar specimens of alloys, Ti-6Al-4V, Ti-16V-2.5Al, Ti-4Al-3Mo-1V, and B120VCA (Crucible's all-beta alloy) were rolled into sheet. All coatings minimized metal loss and contamination during rolling and heat treatment and exhibited lubricating properties by reducing roll pressure requirements under specific temperature and thickness conditions.

Metal loss due to scale formation was eliminated and the skin remaining after removal of the coating was less than that on the uncoated specimens after the loose oxide was removed. The mechanical properties of the alloys are not changed appreciably by the use of coatings. Ti-16V-2.5Al exhibited 5 to 8 ksi higher ultimate tensile strength in both the solution treated and solution treated and aged conditions when the alloy was coated.

AMC TR 60-7-677

September 1960

SUBJECT: TITANIUM SCALE REMOVAL BY CHEMICAL PROCESSES
INVESTIGATOR: M. A. Komertz
CONTRACT: AF33(600)-36858, Syracuse University
ABSTRACT: Aircraft and missile quality hot rolled titanium alloys have been chemically descaled at low temperature (220°F) without degradation of mechanical properties and no hydrogen absorption. Ti-6Al-4V and Ti-2.5Al-16V have been satisfactory descaled in the following low temperature solutions:

- a. 50% NaOH + 5% CuSO₄ · 5H₂O
- b. 50% NaOH + 5% CuSO₄ · 5H₂O + 5% Na₂SO₄
- c. 50% NaOH + 10% CuSO₄ · 5H₂O

Bath temperatures of 200°F and less were investigated with unsatisfactory results. By means of well established processes as well as new experimental processes chemical scale removal has been investigated with respect to temperature, descaling efficiency, the effect on microstructure, hydrogen absorption by the underlying alloy and mechanical properties. Commercial descaling processes investigated were: Virgo Process (Hooker Chemical Company), Molten Sodium Hydroxide, Sodium Hydride (DuPont de Nemours), Temco Electrolytic and 40% aqueous Sodium Hydroxide. All

AMC TR 60-7-677 (Continued)

processes were able to satisfactorily remove the scale from the Ti-6Al-4V alloy with little or no adverse effect on microstructural and mechanical properties. For the Ti-2.5Al-16V alloy, the Virgo, the molten Sodium hydroxide and the Sodium Hydride processes were satisfactory for scale removal at temperatures above 200°F, however, the mechanical properties of the alloy were adversely affected due to descaling, i.e., there was an increase in tensile and yield strength by nearly 200%, a decrease in ductility and there was hydrogen absorption. Low temperature descaling (near 200°F) of this alloy was also studied with experimental baths consisting of aqueous sodium hydroxide, copper sulphate and sodium sulphate in variable amounts.

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